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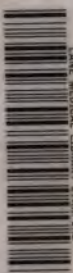
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EST OF CRITICISMS

ON THE

UNITED STATES PHARMACOPŒIA,

SIXTH DECENNIAL REVISION (1880).

PUBLISHED BY

THE COMMITTEE OF REVISION AND PUBLICATION
OF THE PHARMACOPŒIA
OF THE UNITED STATES OF AMERICA
(1880-1890).

PART III.

NEW YORK.
1890.

[This Pamphlet is not for sale.]

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P54
1890
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PREFACE TO PART III.

SINCE the completion of the second part of this Digest, the compiler, Mr. HANS M. WILDER of Philadelphia, has been engaged in collating, from all sources accessible to him, or put at his disposal by the undersigned, such additional material as was deemed worthy of consideration by the next Committee of Revision. These last gleanings could not, of course, be expected to be as numerous or extensive, as those secured for the two parts already issued; but their collection involved a proportionately longer time, since the search for new matter had to be made with constant reference to the text of the Pharmacopœia, as well as to the comments already published.

It is believed that no important paper or publication has escaped notice. At all events, both the compiler and the undersigned have spared no pains in searching for new sources of information. Should it be found, however, that some important contribution has been overlooked, it is suggested that a notice of its title and place of publication, or, if possible, a copy of it, be forwarded to the next Committee of Revision.

This part of the Digest brings the compilation down to the close of the year 1889. Just before going to press, it was possible to insert abstracts of a few important papers which have appeared during this year. Others could only have been made use of at the cost of delaying the publication of this part. They had, therefore, to be neglected.

In quoting authorities, the usual rule has been followed, to name the person or persons appearing as the responsible author or authors of a paper, whether they be individuals or manufacturing firms. In the present part, several publications not previously used have been abstracted; their titles, though more or less abbreviated, will be easily recognized. In a number of instances, corresponding passages from several recently revised European Pharmacopœias (such as the Austrian and the Dutch) have been given for the purpose of completing the information as much as possible.

In the preface to Part II, the undersigned published *two provisional lists*, one giving the titles of articles recommended to be dropped from the Pharmacopœia, and another, enumerating new

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chemicals, drugs, and preparations which had been suggested as proper additions to the official series. Attention is called to the fact that these lists were not announced as the outcome of any official deliberation or vote on the part of the Committee. On the contrary, they are to be considered merely as rough lists, devoid of any official character, though they were prepared on the basis of a variety of reports and records available to the undersigned.

CHARLES RICE,

Chairman of the Committee of Revision, etc.

NEW YORK, *March 10, 1890.*

EXPLANATION OF ABBREVIATIONS IN PARTS I., II. AND III.

- A. —AMERICAN JOURNAL OF PHARMACY.
- B. —AMERICAN DRUGGIST (formerly NEW REMEDIES).
- C. —PHARMACEUTICAL RECORD.
- D. —WESTERN DRUGGIST.
- E. —DRUGGISTS' CIRCULAR.
- F. —EPHEMERIS (Dr. Squibb).
- G. —PHARMACEUTISCHE RUNDSCHAU (New York).
- H. —PHARMACIST (Chicago).
- I. —NEW REMEDIES.
- K. —PHARMACEUTISCHE CENTRALHALLE.
- L. —ARCHIV DER PHARMACIE.
- M. —PHARMACEUTICAL ERA.
- N. —PHARMACEUTICAL JOURNAL AND TRANS. (London).
- O. —PROCEEDINGS OF AMER. PHARM. ASSOCIATION.
- P. —NATIONAL DISPENSATORY, 4th edition.
- R. —COMPANION TO THE U. S. PHARM. (Oldberg-Wall).
- S. —CHEMIST AND DRUGGIST (London).
- T. —U. S. DISPENSATORY, 16th edition.
- U. —REMINGTON'S PRACTICE OF PHARMACY.
- W. —NATIONAL DRUGGIST (St. Louis).
- X. —BECKURTS-HIRSCH: HANDBUCH D. PRAKTISCHEN PHARMACIE.
- Y. —DEUTSCH - AMERIKANISCHE APOTHEKER - ZEITUNG (New York).
- Z. —PHARMACEUTISCHE ZEITUNG (Berlin).
- Un. Ph.—UNIVERSAL-PHARMAKOPÖE (Dr. B. Hirsch).

DIGEST OF CRITICISMS

ON THE

UNITED STATES PHARMACOPŒIA

SIXTH DECENNIAL REVISION (1880).

PART III.

General Remarks.

Adulterations. No need of mentioning them. Fennel. (B. 90, 1).

Alkaloids. Dieterich states that the presence of sugar makes the estimation of alkaloids more difficult. Tschirch has had the same experience. (Z. 89, 599).

Alkaloids-reagent. Kundrat uses vanadin-sulphuric acid, made by dissolving 0.1 Gm. of vanadate of ammonium in 10 C.c. of sulphuric acid. (G. 89, 95. Ch. Zeit., 89, No. 17).

Assays. Should be as simple as possible, approximate results only being aimed at. Fennel (B. 90, 1).

Balsams, Resins and Gum Resins. Acid and ester figure. Dissolve 3 Gm. of the balsam, etc., in 40 Gm. of alcohol, add a few drops of phenolphthalein solution, and add cautiously semi-normal alcoholic solution of potassa, until the red color appears. The number of Mgm. of potassa necessary for every 1 Gm. of substance gives the *acid figure*. To this (titrated) solution add 20 to 30 C.c. of semi-normal alcoholic potassa, heat for one half hour on a water-bath, with frequent shaking, and after cooling, add a little phenolphthalein solution, and rétitrate with semi-normal acid. The number of mgm. of potassa gives the *ester figure*. The sum of the acid and the ester figure gives the *saponification* figure. Kremel (K. 89, 238).

Date for the Pharmacopœia to go into effect. Fennel recommends July 1, 1891 (B. 90, 1).

Nomenclature. Parke, Davis & Co. call attention to several passages in Bentham & Hooker, Genera; Hooker, Flora Indica; Watson, Index; Baillon, Nat. History; and other authors, which seem to indicate that the nomenclature of the U. S. Ph. as to authorities cited, priority, and genus names, needs revision. [Acacia, Alce, Amygdala, Aurantium, Castanea, Catechu, Chirata, Cimicifuga, Cubeba, Hamamelis, Jalapa, Lappa, Macis, Picrotoxinum, Pilocarpus, Pix Burgundica, Pix Canadensis, Prinos, Rhus Toxicodendron, Rubus Idaeus, Stramonium, Taraxacum, Terebinthina

Canadensis, Triticum, Veratrina, Xanthoxylum.] (Drug. Bull., 89, 271).

About the rules governing nomenclature see Rusby (Drug. Bull., 88, 103).

See also remarks by Fennel. (B. 90, 1).

Maximal doses. Of new remedies (A. 89, 488. O. 89, 365. L. 89, 762; 950).

Evaporation. An automatic apparatus for evaporating to a definite weight, and then shutting off the gas, by Ray, see (C. 89, 263).

Extractive of drugs determined. An easily constructed apparatus by Johnson, see (Drug. Bull., 89, 356).

Filters. Economical way of folding. Claassen (A. 89, 74; 159. B. 89, 51; 77. O. 89, 344).—Fessenden (C. 89, 313. Ch. News).

Indicators. Some remarks about differentiation by indicators, by Curtman (Meyer Drug., 89, 24)—See also Allen (E. 89, 175. O. 89, 724. N. 89, May, 902; June, 1028).

Maceration "and" percolation. Lloyd (B. 89, 132. O. 89, 79. D. 89, 159; 236).

Percolation. The committee of the N. Y. State Ph. Association (on Nat. Formulary) recommend to pack the powdered drugs in layers of about half an inch, alternating with sheeted absorbent cotton, claiming that "channeling" will be prevented, and percolation hastened. (E. 89, 186).

See remarks by Fennel (B. 90, 21).

Precipitation. See remarks by Fennel (B. 90, 2).

Pills. Apparatus for determining the solubility, by Meier (Drug. Bull., 89, 226).

Reagents, testing the purity. Krauch (B. 89, 44; 72; 92; 114).

Color reactions of furfural. Udransky (L. 89, 86. C. 89, 364. Zeit. Physiol. Ch.).

Solubility. Correct and incorrect methods of determining. Salzer (G. 85, 209. Z. 85, 523).

A table of solubilities should be based on solutions saturated at the standard temperature (f. i. 20°C.). Fennel (B. 90, 2).

Specific gravity. No need of a uniform method of taking sp. gr. Best taken at 20° C. (68° F.), compared with water at same temperature. See remarks by Fennel (B. 90, 1).

Specific volume. See remarks by Fennel (B. 90, 1).

Standards. Fennel recommends that crude drugs and preparations be standardized. (B. 90, 1).

Synonyms. No need of quoting the official Latin names of the Germ. Pharm. Fennel (B. 90, 2).

Starch paste, which keeps. Mix 50 parts of potato starch and $\frac{1}{16}$ part of biniodide of mercury with 10,000 parts of boiling water; decant the liquid after a while. Gastine (A. 88, 560. O. 89, 425. Rundschau. Drug., 88, 783).

Gelatinized starch. It is directed to be freshly prepared—a remark that it must be used when *cold*, should be added.

Thermometers, history. Browne (E. 89, 127. N. 89, June, 1010; 1030).

How far to carry washing and exhaustion. Proctor determines the point by what he calls a "scale of small residues" (A. 89, 532. N. 89, Oct., 318).

Acacia.

Indian gum substitutes, behavior to reagents (Amrad ; Umrawatti ; Ghatti). Holfert (G. 89, 69. K. 89, 37).

Acidum Aceticum.

A process for its preparation should be given. Its source should be stated. Fennel (B. 90, 21).

Acidum Aceticum Glaciale.

According to Krauch chemically pure acetic acid should possess the following properties: It should be clear and colorless, and of 1.064 sp. gr., 96 p. c.—On evaporating 50 Gm., the residue should not amount to more than 1 Mgm.—10 Gm. diluted to 100 C.c. should not be altered on adding ammonia in excess, nor after the addition of sulphide or oxalate of ammonium.—20 Gm. diluted to 100 C.c. should not show a brown coloration on being mixed with recently prepared hydrosulphuric acid.—10 Gm. diluted with 150 C.c. of distilled water, heated to boiling, and chloride of barium added, should remain clear for several hours.—5 Gm. diluted to 50 C.c. should not be altered by solution of nitrate of silver, acidulated with nitric acid.—5 Gm. diluted with 15 C.c. of distilled water and 3 C.c. of centinormal solution of potassium permanganate, should not fade within 15 minutes. (B. 89, 44.)

Acidum Arseniosum.

Flückiger gives a resumé of the different tests—Marsh ; Berzelius ; Reinsch ; Bettendorff ; Reichardt ; mercuric chloride ; Gutzeit—which latter he considers the most sensitive ($\frac{1}{1000}$ Mgm).

Gutzeit's test is based on the action of arseniuretted hydrogen on a solution of nitrate of silver. Flückiger observes that, if the paper be moistened with a very concentrated solution of nitrate of silver, a yellow spot appears, while with a more dilute solution (1:1) the spot will be black. He further finds, contrary to Reichardt (Part I. 37. B. 84, 33; 34. L. 83, 593), that there is no difficulty in obtaining the color spot by evolving the arseniuretted hydrogen from an alkaline solution (see U. S. Ph. under bismuth), although he admits that the evolution is best done from an acid liquid. As to the strength of the acid liquid he recommends not to use a greater strength than $7\frac{1}{3}$ p. c. (1.036) of hydrochloric acid, or $8\frac{1}{3}$ p. c. (1.055) of sulphuric acid. The silver solution is best obtained by keeping an excess of nitrate of silver in water ; both aluminium and zinc must be absolutely free from arsenic and phosphorus, and the zinc in addition free from sulphur and antimony—the presence of all of which will vitiate the test. (A. 89, 133. G. 89, 71. O. 89, 566. L. 89, 1 to 30. Z. 89, 73).

Gutzeit's test, modified by Siebold. To 5 C.c. dilute hydrochloric acid (1 vol. of sp. gr. 1.16 and 7 of water) and 15 to 20 drops of the liquid to be examined, in a test-tube, add about 1 Gm. of chemically pure zinc, and let the evolved gas strike a piece of white filter-paper, on which a drop of mercuric chloride solution has been allowed to dry: $\frac{1}{100}$ of a Mgm. produces a yellow stain within 15 minutes. The action of sulphur compounds can be guarded against by adding

sufficient solution of iodine to the acid mixture to color it distinctly. (S. 89, Sept., 368).—Siebold also calls attention to the fact that a good deal of the glass tubing contains arsenic. (*Ibid.*, 369).

Klein combines Marsh's and Gutzeit's tests, by inserting layers of powdered nitrate of silver with glass wool in a Marsh's tube. (L. 89, 966).

Ritsert intensifies Gutzeit's test by moistening the paper with an ammoniacal solution of nitrate of silver. The test, carried out according to Gutzeit's method, shows as little as 1000 Mgm.; while according to Ritsert's method 10000 to 20000 Mgm. can be detected. (A. 89, 414. Z. 89, 368).

The ammoniacal silver solution of Ritsert consists of equal volumes of glycerin and ammonia, to which are added a few drops of silver nitrate solution (1:20). The solution of Schmidt is a mixture of equal volumes of silver solution (1:100) and glycerin, with 1 or 2 drops of ammonia. Salzmann considers Schmidt's solution the most sensitive. (K. 89, 524).

It must be completely volatilized on heating in a glass tube. Curtman (Meyer Druggist, 88, 226).

An apparatus for the quantitative estimation of arsenic by Polenske. See (G. 89, 43. Z. 89, 299).

Acidum Carbolicum.

In order to prevent the separation of the acid from a 5 p. c. solution at low temperature, Houssaye adds 5 to 10 p. c. of glycerin. Such a solution may freeze solidly, but no carbolic acid separates. (O. 89, 626. Z. 89, 275. Jour. de Ph. et de Ch.).

Acidum Chromicum.

According to Krauch, pure chromic acid should be in small red, dry crystals—2 Gm. dissolved in 20 Cc. of water should not be altered within 10 minutes on the addition of a few drops of hydrochloric acid and chloride of barium. (B. 89, 44).

Acidum Citricum.

Large colorless, transparent crystals, permanent in the air, efflorescing at a gentle heat, fusing at about 165° C., and charring when ignited. It is soluble in 0.54 parts of water, 1 part of alcohol, and 50 parts of ether. Krauch (B. 89, 44).

Acidum Hydrochloricum.

It should not color chloroform, even after the addition of chlorine water. Curtman (Meyer Druggist, 88, 103).

It should be odorless after dilution with water.—5 Gm. diluted with 50 C.c. of water should not be rendered turbid within 12 hours on adding chloride of barium.—100 Gm. should not contain more than 1 Mgm. of absolute sulphuric acid.—50 Gm. on evaporation should not leave more than 1 Mgm. of residue.—Dilute 10 Gm. with 10 C.c. water, and pour on top of it a layer of 5 C.c. of recently prepared hydrosulphuric acid: no colored ring should be observed within 1 hour.—To 20 Gm. of the same dilution add a slight excess of ammonia and a few drops of sulphide and oxalate of ammonium: no

alteration or dark tint should be observed.—5 Gm. diluted to 25 C.c. with addition of a few drops of a solution of sulphocyanate of potassium, should not acquire a reddish tint.—A few C.c. of this diluted acid added to water, colored faintly blue by iodide of starch, should not destroy the color.—To 5 C.c. of highly diluted solution of starch add a few drops of solution of iodide of potassium, one drop of diluted sulphuric acid and one drop of diluted hydrochloric acid; no blue color should be observed. Krauch (B. 89, 44).

Acidum Hydrocyanicum Dilutum.

The addition of dilute hydrochloric acid, as a preservative, is recommended (Drug. Bull., 89, 336).

Acidum Nitricum.

Test for nitrates in mixtures. To a solution of 0.01 Gm. of resorcin in 6 to 7 drops of sulphuric acid, add 0.02 Gm. of the substance, when a blue color will appear. Goeldner (A. 89, 507. Z. 89, 515).

Estimation of nitrites. Heat the nitrite solution with chloride of ammonium, and measure the nitrogen evolved. Day (B. 89, 67. Jl. Ch. Soc., 88, 422).

Acidum Oleicum.

According to Grandval and Valser, oleic acid is often adulterated with linoleic acid. This may be detected by shaking the acid with an equal volume of solution of soda, when the color of the mixture will be yellow; if the oleic acid is pure, the color is gray. (A. 89, 244; 475. O. 89, 643. Jl. d. Ch. et d. Ph., 89, 232).

Benedikt and Hazura find that oleic acid from vegetable fats always contains linoleic acid. (A. 89, 356; 507. Ch. Repert., 89, 168; 216).

Squibb states that oleic acid is never neutral to test-paper (F. 1244).

Acidum Phosphoricum.

Previous to examining for arsenic acid, Curtman oxidizes any phosphorous acid present by adding solution of permanganate of potassium drop by drop, until it is no longer decolorized. (Meyer Druggist, 88, 149).

Acidum Salicylicum.

Its isomers and homologues. Hesse (O. 89, 265. M. 89, 305).

The presence of cresotic acid may be ascertained by the behaviour of the salicylic acid under hot water. The pure acid does not melt, while in the presence of 20 to 30 p. c. of cresotic acid it will form oily drops. For a method of increasing the p. c. of the latter acid so as to be indicated by the test, see Fischer (A. 89, 353. K. 89, 438. Z. 89, 329).

Acidum Sulphuricum.

After dilution with much water, it should not color blue a dilute solution of iodide of potassium and starch. Curtman (Meyer Druggist, 88, 171).

Test for Lead. Carefully pour some hydrochloric acid upon a few C.c. of sulphuric acid, contained in a test-tube. A white turbidity at the point of contact shows the presence of lead (*Ibid.*, 88, 71).

Test for Purity. 10 Gm. should, on evaporation, leave no residue. If to 100 Gm. there are added 10 volumes of pure water, colored by 1 drop of indigo solution, the addition of 100 C.c. more of water should not cause a discharge of color (this test detects 1 Mgm. of nitric acid in 1 kilo of sulphuric acid).—To 60 C.c. of water add 15 C.c. of the acid and 1 drop of decinormal solution of permanganate of potassium (1 C.c. of which is equal to 0.0056 Gm. of iron): a distinct red tint should be produced. Diluted with water and mixed with ammonia in excess, it should not acquire a greenish tint on the addition of sulphide of ammonium, nor be rendered cloudy by oxalate of ammonium.—On diluting 2 Gm. of the acid with 30 C.c. of water, adding 3 to 4 Gm. of solution of potassa and 10 to 12 drops of Nessler's reagent, no distinct yellow or reddish-brown color should be observed. (This test shows 1 Mgm. of ammonia in 100 Gm. of acid). Krauch (B. 89, 72).

Acidum Tannicum.

A solution of 1 part in 5 parts of boiling water should remain clear on addition of a double volume of alcohol (absence of gum and dextrin); also on addition of a double volume of cold water (absence of resin). Curtman (Meyer Druggist, 88, 172).

Acidum Tartaricum.

Assay. Haidenhain finds that acetate of potassium decomposes the bitartrate, which can be prevented by adding 2 parts of free acetic acid for every 1 part of tartaric acid combined with potassium. Then add alcohol 10 parts to 1 part of the aqueous solution. See further (B. 89, 96. Zeit. Analyt. Ch., 88, 704).

Aconitum.

Squibb's physiological Test. (See F. 125; 503. A. 84, 277). Thompson points out that 1:600 does not suit all persons; 1 part diluted to 500 would be the limit for many. Since this test is merely a comparative one, one has to find out his own limit of dilution, and use this throughout.

Assay. Thompson makes use of the above test, the amount of dry extract and Mayer's reagent. Exhaust 10 Gm. with alcohol to make 100 C.c., 10 C.c. of which are evaporated and dried at 105° C., which will represent 1 Gm. of the root.—Distil off the alcohol from 50 C.c. (=5 Gm. of the root) of the above tincture by a steam bath; add 10 drops of 5 p. c. sulphuric acid and water to make 10 C.c., and finally Mayer's semi-normal volumetric solution, as long as a precipitate occurs.—10 C.c. (=1 Gm. of the root) are diluted with water until 1 fluidrachm produces the familiar impression.

Thompson estimates the aconitine by dissolving 0.020 Gm. in 100 C.c. of water (containing 25 of alcohol and 2 minims of diluted sulphuric acid, 1:5), which solution is diluted until 1 fluidrachm gives the normal sensation. On examining in this way the various aconitines of commerce, he found that Gehe & Co's amorphous was 45

times stronger than the root; Duquesnel's crystallized 300 times; Merck's crystallized 350 times; Gehe & Co.'s crystallized 480 times—results which are decidedly higher than Squibb's own (see F. 135) [Merck's ordinary 8 times; his pseudo-aconitine 83 times; Duquesnel's, 111 times.] (Drug. Bull., 89, 108).

The best menstruum for exhausting aconite is a mixture of 3 volumes of alcohol (84 p. c.) and one of chloroform. 20 C.c. of the tincture prepared with this menstruum contained 0.076 Gm. of alkaloid, against 0.066 Gm. in the tincture made with alcohol of 84 p. c., and against 0.034 Gm. in a tincture made with chloroform. Wright (A. 89, 536. N. 89, Nov., 376).

Adeps.

Pattison modifies Becchi's nitrate of silver test (see Part I., 113. B. 88, 110) by adding an alcoholic solution of the silver salt to an ethereal solution of lard (shows as little as 5 p. c. cottonseed oil). He also gives the iodine absorption test and the sp. gr. (B. 89, 72. Jl. Soc. Ch. Ind., 89, 30).

Ritsert recommends to boil it for 5 to 8 minutes with an equal volume of a 2 p. c. alcoholic solution of nitrate of silver (acidulated with 0.5 p. c. of nitric acid). Pure lard will remain clear and colorless; cotton seed oil yellow to grayish green and brown; mucilaginous matter reduces the silver, and chloride of sodium precipitates it. (L. 89, 233. Z. 89, 73).

Bradford recommends his subacetate of lead test (previously used for olive oil), as a reliable test for cotton seed oil. Equal weights of lard and solution of subacetate of lead are heated in a water-bath in a test-tube until fluid, when the tube is shaken. After cooling, the red color will indicate cotton-seed oil (C. 89, 383.)—See also under *Oleum Olivæ*.

Lard which reduces silver nitrate, and absorbs more than 66 p. c. of iodine, is to be rejected (K. 89, 199).

Melting point. Kohlmann ascertains it after the principle of Carpi's spindle test for olive oil (L. 89, 828). The spindle test see (B. 84, 44).

Adeps Benzoinatus.

Williams recommends the old process of mixing an ethereal solution of benzoin with castor oil, and evaporating the ether. (O. 89, 417. D. 89, 81).

Utescher dissolves 1 part of benzoin-benzoic acid in 100 parts of lard. (A. 89, 246. O. 89, 418. Ap. Zeit., 89, 280).

Aether Fortior.

Test for aldehyd. Crismer strongly recommends Nessler's reagent, which gives a yellowish or reddish-brown to black precipitate, which differs from that produced with ammonia, by being insoluble in solution of cyanide of potassium. (Z. 89, 384).

Sulphur is detected by shaking the ether with bright mercury, when the latter will assume a dull color. De Koninck (B. 89, 96. Z. 89, 22).

Ten Bosch states that Koninck's test for sulphur (with metallic mercury) is unreliable, because the blackening may also be caused

by peroxide of hydrogen, often present in ether. (E. 89, 131. Z. 89, 222).

Test for purity. Sp. gr. 0.722 to 0.720. Boils at 34 to 36° C. On slowly evaporating 50 Gm., no residue should be left.—30 C.c. poured upon 5 Gm. of pure potassa, and kept for a few days in a dark closet with occasional agitation, should not separate brown flakes.—To 15 C.c. of the ether in an absolutely dry test tube add a piece of sodium the size of a pea; the sodium should not lose its metallic lustre within 6 hours. Krauch (B. 89, 73).

Test for Vinyl Alcohol, which is reported to be a constant concomitant of ether unless specially freed from it, and then kept from air and light. Reagent: Mix 4½ vol. of a saturated aqueous solution of potassium bicarbonate, and 1 vol. of a saturated aqueous solution of mercuric chloride, and filter. On shaking ether with this solution [about 3 vol. ether and 1 vol. reagent?], the aqueous layer will become opalescent and opaque, after the mixture has stood a short time, and eventually throw down a white precipitate, if vinyl alcohol was present. Thümmel (B. 90, 13. L. 89, 961).

Aether Aceticus.

"Should not measure less than 9 C.c."—Curtman requires 9.3 C.c. (Meyer Druggist, 89, 97).

Solubility, according to Allen, is 1 meas. in 26 at 0° and 1 in 24 at 15° C. The color of litmus changes upon the volatilization of the ether. Fennel (B. 90, 21).

Alcohol.

Mixed with 2 or 3 volumes of distilled water, it should appear clear after the numerous air bubbles have disappeared (Ph. Hung.)—Should be miscible with water in all proportions without becoming turbid. Curtman (Meyer Druggist, 88, 74).

Before applying the nitrate of silver test, the silver solution should be tested with chemically pure alcohol. Hirsch (Un. Ph. II., 614).

Test for amylic alcohol. To 5 C.c. of alcohol add 2 drops of an aqueous solution of furfural (0.5 p. c.), and cautiously pour in 5 C.c. of concentrated sulphuric acid, so as to form a layer beneath, cover and cool the mixture to 60° C. or below. In the presence of amylic alcohol a red color, turning to violet, will appear. Udransky (G. 89, 173. Z. 89, 370. L. 89, 465).

Test for aldehyd. A mixture of a solution of fuchsine in water (1:1000) with a solution of bisulphite of sodium (30° B.), containing in 30 C.c., 10 C.c. hydrochloric acid, is clear. On shaking 2 C.c. of alcohol with 1 C.c. of the above mixture, a purplish-red color will appear in the presence of aldehyd. Guyon (L. 89, 419).—Test for aldehyd with Nessler's reagent, see under Aether.

Test for furfural, by colorless aniline and hydrochloric acid; or by xylydine and acetic acid.—for *organic bases* by Kjeldahl's ammonia method. See (B. 89, 131).

Absolute Alcohol, test of strength. A small quantity of gelatinized starch is poured over a large watch crystal, so as to produce a thin film, which is then thoroughly dried at 100° to 120° C. Add sufficient iodine to alcohol to make a light brown solution, and pour it over the starch film—no blue color should be produced. Nagelvoort (Drug. Bull., 89, 335).

See remarks by Fennel (B. 90, 21).

Test for water. Alcohol of more than 98 p. c. (vol. ? weight ?) is miscible in all proportions with bisulphide of carbon; at 98 p. c. only with an equal vol. Barfoed.—A solution of 1 C. c. of benzol in 2 C. c. of absolute alcohol requires the addition of 10 C. c. of alcohol (70.9 p. c. vol.) before a permanent cloudiness appears. Hager.—Other tests as: Sulphate of copper; carbonate of potassium; paraffin oil; citromolybdic acid; anthraquinon, see (B. 89, 131).

Ammoniacum.

Acidity; ester and saponification numbers. Dieterich, see (A. 89, 357. Ph. Post., 89, 336).

Ammonii Benzoas.

A dilute aqueous solution, acidulated with nitric acid, should not give a precipitate with barium or silver nitrate. Curtman (Meyer Druggist, 88, 196).

A formula should be given: ammonium carbonate 157 parts, water ammonia 200 parts or q. s., benzoic acid 122 p., water 400 p. See Fennel (B. 90, 2).

Ammonii Carbonas.

The statement of the U. S. Ph. that 2.616 Gm. should require 50 C. c. of the volum. solution of oxalic acid, which has been controverted by Maisch and Squibb (see Part I., 26), is according to Behringer's investigations correct for the *officinal* salt which is NH_4HCO_3 - $\text{NH}_4\text{NH}_2\text{CO}_2$, while of the ordinary commercial salt ($2 \text{NH}_4\text{HCO}_3$ - $\text{NH}_4\text{NH}_2\text{CO}_2$) 2.95 Gm. would neutralize 50 C. c. of oxalic acid (A. 89, 500).

The test for strength is theoretically correct, though in practice it may vary. Fennel (B. 90, 2).

Ammonii Chloridum.

Purification: sat. sol. is supersaturated with water of ammonia, set aside 48 hours, then filtered, and evaporated. Test for iron cannot be too rigorous. Fennel (B. 90, 2).

Ammonii Iodidum.

Falk decolorizes it by placing a lump of carbonate of ammonium in the bottle, until the salt has regained its whiteness. (A. 89, 464. C. 89, 303).

Fennel recommends putting the colored salt under a bell-glass with a dish containing a little water of ammonia (B. 90, 2).

Ammonii Valerianas.

The commercial salt being acid, it should be neutralized with ammonia. Fennel (B. 90, 2).

Amyl Nitris.

On metameres, see Dunstan and Williams (A. 89, 148. B. 89, 148. O. 89, 623. N. 88, Dec., 487).

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red color in presence of nitrites (A. 89, 470. Z. 89, 429). See also under Acidum Nitricum.

Aqua Ammonia.

Lunge and Wiernik have calculated a new table of sp. gr. and p. c. (A. 89, 516. B. 89, 139. Z. 89, 314. Zeit. Angew. Ch., 89, 181).

Grüneberg likewise (A. 89, 516. C. 89, 178. Ph. Weekbl.)

Test for pyridine. On adding nitric acid in excess, it should remain colorless. Krauch (B. 89, 114).

[Aqua Medicata.]

Falk recommends paper pulp (Meyer Druggist, 88, 244. B. 89, 8).—Stein likewise (A. 89, 380).

Purified wood pulp is also suggested (B. 89, 18).

Strength. Davies suggests to assay them by Hübl's iodine absorption method, shaking up a known quantity with chloroform, and digesting the chloroform solution with Hübl's reagent (A. 89, 309. N. 89, April, 824).

Aqua Chlorig.

When shaken with chloroform or carbon disulphide, these reagents should not become colored, even transiently; a brownish color indicating bromine, and a violet tint, iodine. Curtman (Meyer Druggist, 89, 20).

Aqua Destillata.

On acidulating 100 C.c. of water with diluted sulphuric acid, and then adding 1 C.c. of solution of starch and chloride of zinc, no blue color should be developed within 10 minutes. Ph. Germ. Com. (B. 89, 112. L. 89, 341).

Argenti Nitras.

A solution of 0.5 Gm. of nitrate of silver in 0.5 Gm. of water, shaken for a few minutes with 20 C.c. of absolute alcohol, should remain clear.—If 2 Gm. are dissolved in 60 C.c. of water, heated to 70° C., precipitated by the gradual addition of hydrochloric acid, and filtered while still warm, the filtrate, evaporated to dryness and ignited at a low red heat, should leave only traces of a residue. Krauch (B. 89, 73).

Asclepias.

Analysis by Quackenbush (A. 89, 113. O. 89, 445).

Aspidium.

The chemistry of filicic acid by Dacomo, see (A. 89, 144. O. 89, 431. Jl. Ch. Soc., 88, 521. 89, 54).

Atropina Sulphas.

The more hyoscyamine sulphate it contains, the handsomer it crystallizes. Schmidt (K. 89, 591).

Balsamum Peruvianum.

Acidity; ester and saponifying number, Dieterich, see (A. 89, 357. Ph. Post, 89, 336).

Balsamum Tolutanum.

Acidity; ester and saponifying number, Dieterich, see (A. 89, 357. Ph. Post, 89, 336).

Belladonnæ Folia.

A good standard would be 0.4 p. c. of alkaloid. Parke, Davis & Co. state that the average obtained in 1886 was 0.46 p. c.; in 1887, 0.44 p. c.; in 1888, 0.31 p. c.; in 1889, 0.25 p. c.; they also found two samples assaying as high as 0.6 and 0.7 p. c. (Drug. Bull., 89, 300). They recommend for assay a modification of Lyons' method (Part I., 55).

Belladonnæ Radix.

The transverse section, moistened with iodine solution, shows yellow wood bundles on a blue ground (distinction from burdock root).

Schmidt states that the length of time the root is kept has very little influence on the state of the alkaloid, but the time of growth is an important factor. No atropine was found in two-year-old roots, while in roots of one year both atropine and hyoscyamine were present (E. 89, 272. K. 89, 590).

Simonson has examined the roots of the American market, and found that in 35 samples the p. c. of alkaloids runs from 0.220 to 0.776, which certainly would indicate a necessity for requiring a lowest limit (O. 89, 121. B. 89, 150. D. 89, 239. C. 89, 216).

A good standard would be 0.5 p. c. of alkaloid. The average obtained in 1886 was 0.58 p. c.; in 1887, 0.52 p. c.; in 1888, 0.57 p. c.; in 1889, 0.55 p. c. For assay, a modification of Lyons' method for the leaves (Part I., 55) is recommended. Parke, Davis & Co. (Drug. Bull., 89, 302).

The best menstruum is a mixture of 4 volumes of alcohol (84 p. c.) and 1 volume of chloroform. 20 C.c. of the tincture prepared with this menstruum contained 0.040 Gm. of alkaloid, against 0.030 Gm. in the tincture prepared with alcohol (84 p. c.), and 0.011 Gm. in that prepared with chloroform. Wright (A. 89, 356. N. 89, Nov., 376).

Benzoinum.

Acidity; ester and saponification number, Dieterich. See (A. 89, 357. Ph. Post., 89, 336).

Bismuthi Subcarbonas.

Nagelvoort recommends Bettendorff's test for arsenic as much more sensitive than that of the Pharmacopœia, the latter not being able to discover the presence of 1 p. c., while Bettendorff's test discovers 0.02 p. c. (Drug. Bull., 88, 93).

Bismuthi Subnitras.

Its solubility in water is much increased by the presence of calcium

salts, hence "hard" water dissolves more of it than soft water. Janecek (Ph. Zeit. Russl., 89, 349).

Bromum.

Sp. gr. 2.970. Bromine becomes solid at -24.5° C.—On shaking the aqueous solution with ferric chloride and chloroform, the latter will become purple in the presence of iodine.—When dissolved in solution of soda, no yellow drops should separate, nor any odor of bromoform be noticed. Curtman (Meyer Druggist, 88, 274).

Calcii Chloridum.

Completely soluble in 10 parts of absolute alcohol.—When the salt is boiled with solution of soda, no vapor of ammonia should be given off, nor turmeric paper be reddened.—Lime water, added to the aqueous solution (1:50), should produce no alteration on longer standing. Krauch (B. 89, 92).

Calcii Hypophosphis.

Test of identity. To an aqueous solution of the salt add test-solution of molybdate of ammonium (containing nitric acid), and then a few drops of sulphurous acid, when a blue coloration or precipitate is formed. Millard (A. 89, 129. O. 89, 528. N. 89, January, 585). The molybdic solution is made by dissolving 1 part of molybdic acid in 4 parts of ammonia, filtering, and pouring into 15 parts of nitric acid (1.20). (N. 88, June, 995.)

Assay. To an aqueous solution add diluted sulphuric acid and sufficient permanganate of potassium to produce a permanent tint, heat awhile, and retitrate with oxalic acid.—To an aqueous solution add hydrochloric acid and a saturated solution of mercuric chloride, heat for one hour, filter and wash the precipitate of mercurous chloride, the weight of which, multiplied with a factor, gives the amount of hypophosphite of calcium.—Oxidize the aqueous solution by bromine, add a neutral solution of chloride of calcium and phenolphthalein, and titrate with normal solution of soda. Moerck (A. 89, 329 to 332; 386).

The test for soluble *phosphate* in the U. S. Ph. becomes unreliable because most of the commercial hypophosphite contains sulphate. Moerck proposes the following requirement: The addition of ammonia to an aqueous solution of the salt, in sufficient quantity to produce an alkaline reaction, should yield no precipitate (A. 89, 391).

Calx.

Soluble in 780 parts of water at 15° C. Curtman (Meyer Druggist, 88, 277).

Calx Chlorata.

Should freedom from arsenic be insisted upon?

Schroeter finds that the average strength of commercial chlorinated lime is 29.83 p. c. (A. 89, 13. O. 89, 522).

Calx Sulphurata.

The composition might be definitely stated. According to the

test for strength the formula would be $3\text{CaS} + \text{CaSO}_4$. Fennel (B. 90, 2).

Cantharides.

Cantharides, previously extracted with ether, may be recognized by exhausting 5 Gm., each, of good flies and suspected flies, separately, with ether. The ethereal tincture from good flies has a yellow, faintly greenish color, and leaves on evaporation 10 p. c. of a syrupy greenish-yellow extract. The tincture from the extracted flies is nearly colorless, and leaves a small quantity of brown extract (Z. 87, 622).

A good standard would be 0.7 p. c. of cantharidin. Parke, Davis & Co. (Drug. Bull., 89, 335).

Assay. Digest the fine powder with a 10 p. c. solution of soda, acidulate with hydrochloric acid, and exhaust with boiling chloroform. Evaporate, wash with bisulphide of carbon, dissolve in chloroform, and evaporate. Nagelvoort (L. 89, 757).

According to Baudin, there exist in the market cantharides extracted by a menstruum, containing sulphuric acid, in which case the ash will contain an undue amount of sulphate. He assays by first exhausting with chloroform, containing 2 p. c. of hydrochloric acid. The cantharidin is present partly in the free and partly in the combined state; usually 1 p. c., which consists of about 0.72 p. c. free and 0.3 p. c. combined cantharidin (A. 89, 21. O. 89, 498. Z. 88, 739).

Carbonei Bisulphidum.

Sp. gr. 1.268.—The odor should not be fetid.—A test for selenium might be added. Curtman (Meyer Druggist, 88, 278).

Purification by lime water. Singer (B. 89, 64. O. 89, 535. J. Soc. Ch. Ind., 89, 96).

Cera Flava.

The solubility in chloroform—11 parts—is erroneous; it should be from 33 to 35 parts.—The sp. gr. should be not below 0.962.—In giving the melting point, the Ph. should state the method, because the temperature varies accordingly. Parke, Davis & Co. (Drug. Bull., 89, 374).

Test for *paraffin*. Hager subjects the wax to sublimation, and dissolves the sublimate in chloroform, which is then further tested. See (A. 89, 564. E. 89, 244. K. 89, 565).

Horn's test for resin (Part II., 208) is, according to Dieterich, worthless, because it scarcely indicates the presence of 20 p. c. of resin (K. 89, 253).

Charta Sinapis.

Dieterich states that a good mustard paper should contain in 100 square Cm. (about 16 sq. inches) at least 1.5 Gm. of mustard flour; which should give off at least 1 p. c. of the volatile oil of mustard (L. 89, 413). For the assay see (O. 87, 29. L. 87, 496; 565).

Chloral.

Test of identity. Schwarz heats the suspected liquid with an excess of resorcin and a few drops of solution of soda to boiling, when

the liquid will assume a yellowish-red color with a yellowish-green fluorescence (B. 89, 13. Zeit. Ang. Ch., 88, 668. Z. 88, 419).

Chloroformum Purificatum.

Hirsch purifies chloroform by allowing the crude article to stand several days with chloride of calcium, then pouring off and distilling from a perfectly dry retort (Z. 89, 584).

It should not liberate iodine from a solution of pure iodide of potassium, which would be indicated by a violet color. Curtman (Meyer Druggist, 89, 21).

In the directions, it should read: "Distil, by means of a water-bath 'containing water at the temperature of 160° F.,' into a well-cooled receiver" . . . "Continue the distillation, until near dryness, at a temperature not exceeding 150° F." According to Allen, the boiling point, 59.8° C., indicates the presence of 0.5 p. c. of alcohol. Iodide of potassium will not react for free chlorine. Fennel (B. 90, 20).

Preservation. Schacht finds that the presence of 0.058 p. c. of amylic alcohol preserves it. He remarks further that a pure alcohol will always insure a pure chloroform (L. 87, 867).

Test of identity. With resorcin, see under Chloral.

Chloroformum Venale.

"Acetone" chloroform, manufacture. Sadtler (A. 89, 321).

Roessler suggests that the "commercial" chloroform be dropped, since the "purified" is at present but little higher in price, and the commercial, at times, is exceedingly impure (G. 89, 33. O. 89, 616).

Cimicifuga.

Analysis by Rademaker (C. 89, 9. O. 89, 464).

Cinchona.

Extraction. A bark of *C. succirubra*, yielding 7.592 p. c. of total alkaloids, of which 2.127 was quinine, on being exhausted with boiling water, yielded 6.442 of total alkaloids and 1.585 p. c. of quinine; alcohol of 90 p. c. extracted 6.597 and 1.827 p. c.; water, acidulated with hydrochloric acid, extracted 5.868 and 1.338 p. c. respectively. The acid menstruum extracts much more readily, and the resulting extract is more readily soluble; besides, the natural flavor is better. Landrin (S. 89, April, 532).

Assay. Landrin finds that both soda and lime are essential for the complete extraction of the alkaloids. 300 Gm. of finely powdered bark are mixed with 75 Gm. of lime and 75 Gm. of solution of soda (40°B), petroleum is added and the mixture heated to 100°C. for twenty minutes with vigorous shaking. Repeat the treatment, extract with water acidulated with sulphuric acid, heat to boiling, and neutralize cautiously with ammonia, when on cooling about 90 p. c. of the alkaloids will be deposited. Precipitate the mother liquor with soda, convert all into sulphate, and weigh (B. 89, 196. Comptes Rendus., 108, 750).

Tschirch states that the phloëparenchym is the seat of the alkaloids (K. 87, 477).

3½ per cent. of the peculiar alkaloids. Cultivated barks, especially *succirubra*. Pharm. Austr.

Pharm. Austr. uses Mayer's method of assay, modified by Kremel. See p. 48, Part I. of "Digest." Instead of dil. sulph. acid, it uses 1 part tartaric acid for every 10 parts of bark represented in the alcoholic extraction, and ether instead of chloroform. The result is stated by Schneider to be quinine and its isomers, with a little cinchonidine.

5 to 6 p. c. alkaloids. Only from *succirubra*. Pharm. Neerl.

Boil 10 Gm. of the powdered bark, (*succirubra* cultivated) and 15 Gm. of slaked lime with 168 Gm. of alcohol (0.833), until the red color of the liquid is turned more or less yellow. Allow to cool, and add alcohol to restore the original weight. Shake well, filter; to 84 Gm. of the filtrate add 3 Gm. dil. hydrochloric acid (1.062), and 10 Gm. water, and evaporate on a water-bath with frequent stirring until 15 Gm. remain. To the still hot liquid add carbonate of sodium, until it is only slightly acid. To the filtered and cooled liquid add soda solution to distinctly alkaline reaction, and shake well, first with 20 C.c., and then with 10 C.c. of chloroform, until no more alkaloid is taken up. Distil off the chloroform, and dry the alkaloids for an hour at 100° C., when they should weigh 250 to 300 Mgm.—Heat the alkaloids, dissolved in 10 Gm. of water and neutralized by hydrochloric acid, with an equal weight of tartrate of sodium; on cooling a little, the tartrate will separate, which, when washed and dried at 100° C. should amount to 55 p. c. of the alkaloids. Dissolve 100 Mgm. of the tartrate in 40 C.c. water by means of a few drops of dil. sulph. acid; of this mix 4 C.c. with 2 or 4 drops of chlorine water and 2 drops of ammonia, when a distinct green color should appear. Pharm. Neerl.

Colchici Radix.

A good standard would be 0.5 p. c. of colchicine. The average of a number of assays was 0.52 p. c., with a minimum of 0.4 p. c. and a maximum of 0.65 p. c.

Assay. Macerate it with modified Prollius' fluid (250 C.c. of ether, 80 C.c. of chloroform, 25 C.c. of alcohol, and 10 C.c. of ammonia), pour off a fractional part of the clear liquid, evaporate the chloroform and ether, take up with water containing 3 p. c. of sulphuric acid, and titrate with Mayer's solution, calculating the result according to Lyons' method (A. 86, 585). Parke, Davis & Co. (Drug. Bull., 89, 302).

Colchici Semen.

A good standard would be 0.7 p. c. of colchicine—the maximum and minimum results of a number of assays being 0.9 and 0.5 p. c. respectively. Parke, Davis & Co. (Drug. Bull., 89, 302).

Collodium.

It can be obtained perfectly transparent by shaking it with quartz sand. Kranzfeld (A. 89, 415. Ph. Zeit. Russl., 89, 392).

Collodium cum Cantharide.

Grazer proposes to make it with cantharidin. Exhaust the cantharides with chloroform, distil off, and remove the fat with bisulphide of carbon or benzin (A. 89, 439. O. 89, 104).

Conium.

A good standard would be 0.6 p. c. of conium. Lyons' method of assay (Pharm. Assaying, p. 85) gives uniform results. The powdered drug is extracted by benzin in the presence of ammoniated alcohol. A fractional part of the clear fluid is shaken with water acidulated with sulphuric acid, the aqueous solution is separated, rendered alkaline by potassium carbonate, and taken up by benzin, which is separated and shaken with a 1 p. c. hydrochloric acid; the aqueous solution is separated and titrated with solution of sodium phospho-tungstate. Parke, Davis & Co. (Drug. Bull., 89, 303).

Copaiba.

Acidity; ester, saponification figures. Dieterich (A. 89, 357. K. 89, 253. Ph. Post., 89, 336). He finds the acidity of East India (gurjun) balsam 6 to 7; Para, 52 to 53; Maracaibo, 73 to 86.

Creasotum.

The sp. gr. should not be below 1.07 (pure creasote 1.089 and pure guaiacol 1.117).—Mixed with 10 volumes of an alcoholic solution of potassa (1:40), creasote is after some time converted into a crystalline mass; but if carbolic acid be present it retains its fluidity.—On mixing 1 C.c. with 2 C.c. of benzin and 2 C.c. of cold saturated baryta water, the benzin must not acquire a blue or muddy color, nor the aqueous solution a red color.—On mixing 4 C.c. of water, 4 C.c. of solution of soda (15 p. c.) and 2 C.c. of creasote, the mixture must be clear and of a light yellow color. Brandes (B. 89, 75; 112. G. 89, 173. O. 89, 629. L. 89, 111; 351).

Crocus.

"It should not color the water *immediately* after immersion."

Cubeba.

Immature cubebs, see Lowe (A. 89, 117).

Cuprum.

Test for copper. Add to 2 C.c. of a cold saturated solution of bromide of potassium, 1 C.c. of concentrated sulphuric acid, shake and add 2 to 3 drops of the suspected liquid, when a carmine color will appear, which disappears on diluting with water. Denigés (A. 89, 289. B. 89, 110. O. 89, 559. Bull. Bordeaux).

Cupri Sulphas.

Test of purity. Dissolve 2 Gm. in 20 C.c. of water, precipitate by hydrosulphuric acid, filter, evaporate and ignite, when only traces of residue should be left (0.07 p. c.). Krauch (B. 89, 92).

Cydonium.

Bruised with water, it evolves an odor of bitter almond (X. II., 419).

Digitalis.

Broeker found 1 p. c. digitalin in the parenchym of the leaf, and in the stalk only 0.2 p. c. The U. S. Ph. ought therefore to insist upon the removal of the stalks (B. 88, 10. Nieu Tijds.)

Dulcamara.

The taste of the bark is bitter, that of the woody portion sweet. Ph. Germ.

Emplastrum Hydrargyri.

Hecker extinguishes the mercury with an equal part of lanolin (Z. 89, 194).

Erythroxyton.

A good standard would be 0.6 p. c. of cocaine. For method of assay see under *Belladonnæ Folia*. Parke, Davis & Co. (Drug. Bull. 89, 303).

Assay. Mix the powder with water and magnesia, dry at 60°C., exhaust with ether, distil off, exhaust residue with a dilute (2 p. c.) hydrochloric acid, render alkaline with ammonia, take up with ether, etc. Van d. Marck (A. 89, 294. B. 89, 147. O. 89, 472. Z. 89, 282).

Culture in India and assay of the East India leaves; see Warden (A., 89, 358; 580. Ch. News, 89, 249; 260; 273. S. 89, Sept. 468).

An interesting article on the commercial varieties, see (Z., 89, 163).

Euonymus.

Analysis by Cassaday (A. 89, 284. O. 89, 494).

[Extracta Solida.]

By the freezing process. History from 1799, see (S. 89, Sept., 471).

Standardized extracts. Duncan points out that some means should be devised, which will prevent the alteration in the consistence of these extracts. Of necessity, standardized extracts must vary in consistence according to the alkaloidal strength of the crude drugs: the stronger the drug, the thinner the consistence of the extract. He suggests the advisability of following the idea of the "abstracts" of the U. S. Ph.: evaporating to dryness and to make up for the loss of weight with milk sugar, or some other inert powder (for instance, kaolin). (E. 89, 102. O. 89, 377. N. 89, Febr'y, 625; 643. S. 89, Febr'y, 156).

Assay. Assay of narcotic extracts by treating with acetate of lead, dilute sulphuric acid, chloroform, ether and centinormal acid by Itallie (A. 89, 134. O. 89, 372. Ap. Zeit., 89, 124). See under *Extractum Aconiti*.

Beckurts states that the assay of extracts is rendered uncertain by the chlorophyll, which is also extracted by chloroform; he removes it with baryta water. Dissolve 5 Gm. of the extract in 50 Gm.

diluted alcohol (0.892), add baryta water in excess, so as to make 150 C.c., allow to deposit, filter and precipitate excess of baryta by carbonic acid gas. 75 Gm. of the filtrate (= 2.5 Gm. of extract) are evaporated to a syrupy consistence and assayed in any convenient manner (K. 89, 577. Z. 89, 581).

Lloyd recommends to moisten the powdered drug, previous to percolation, by sprinkling it with 3 fluid ounces of water to each pound, and after ten to twelve hours to moisten with the proper menstruum, etc. (O. 89, 81. B. 89, 148. G. 89, 165. C. 89, 223).

Sick states that each extract appears to have its special line (or lines) of absorption in the spectrum (S. 89, April, 533).

[**Extracta Fluida.**]

A table of the amount of dry extractive obtained from several fluid extracts by Thompson. See (Drug. Bull. 88, 169).

Manufacture on a large scale. Webber (Drug. Bull. 87, 247).

Extractum Aconiti.

It should be 5 times as strong as the root. P. D. & Co. (Drug. Bull. 87, 195).

Assay. Triturate 5 Gm. of the extract with 10 drops of diluted sulphuric acid (1 : 20) and sufficient water to make 50 C.c.; after 2 hours add 25 C.c. of solution of acetate of lead (1 : 10). Filter off 50 C.c., add 10 C.c. of diluted sulphuric acid, filter, render alkaline with ammonia and extract the alkaloid by shaking with chloroform in 3 successive portions of 50 C.c. each. Distil off, take up with 5 C.c. of diluted alcohol, add cochineal tincture, and titrate with centi-normal hydrochloric acid. 1 C.c. of the acid indicates 0.00533 Gm. of aconitine. Itallie (A. 89, 134. O. 89, 372. L. 89, 185).

Extractum Aconiti Fluidum.

A good standard would be 0.75 p. c. of alkaloid. 10 C.c. of the fluid extract should require 2.8 C.c. of Mayer's solution for complete precipitation. Parke, Davis & Co. (Drug. Bull., 87, 194).

Extractum Belladonnæ Alcoholicum.

A good standard would be 1.75 p. c. of atropine. Parke, Davis & Co. (Drug. Bull., 87, 195; 89, 302).

Assay. Similar to the one mentioned under extract of aconite. 1 C.c. of the acid indicates 0.00289 of atropine and hyoscyamine. Itallie (A. 89, 134. O. 89, 372. L. 89, 185).

Extractum Belladonnæ Fluidum.

A good standard would be 0.5 p. c. of atropine. To 10 C.c. add a few drops of diluted sulphuric acid, evaporate off the alcohol, wash the residue with ether, take up with water, and extract the alkaloid with a mixture of ether and chloroform, after the addition of an alkali. Parke, Davis & Co. (Drug. Bull., 87, 194. See also 89, 302).

Extractum Cannabis Indicæ Fluidum.

Ten C.c., evaporated to complete dryness at 105° C., should leave a residue of 1 Gm. Parke, Davis & Co. (Drug. Bull., 87, 194).

Extractum Cinchonæ.

It should be 5 times as strong as the bark. Parke, Davis & Co. (Drug. Bull., 87, 195).

Assay. See under Cinchona, 84 Gm. of the filtrate (=3 Gm. of the extract), should yield 270 to 330 Mgm. of alkaloids, equal to 9 to 11 per cent. Pharm. Neerl.

Extractum Cinchonæ Fluidum.

A good standard would be the lowest official one of the bark, 2 p. c. of anhydrous quinine. Parke, Davis & Co. (Drug. Bull., 89, 194).

Stoeder gives the following improvement of De Vrij's formula: The powdered cinchona, of known alkaloidal strength, is mixed with the calculated amount of diluted hydrochloric acid (73 parts of absolute acid to 300 parts of alkaloids), glycerin and 3 times its weight of water, transferred to a percolator, and percolated, after 24 hours maceration, with distilled water until almost the whole amount of cincho-tannates has been obtained. Evaporate the percolate at 70° C. until the weight of the fluid extract equals the weight of the bark (Z. 89, 221. K. 89, 325).

Mix 5 Gm. of the fluid extract with 40 Gm. water and sufficient solution of soda (1.33) to a distinctly alkaline reaction; shake, and extract with 20 and then with 10 C.c. of chloroform. The alkaloids, dried at 100° C., should weigh 200 to 225 Mgm. = 4 to 4.5 p. c. Pharm. Neerl.

Extractum Colchici Radicis.

It should be 5 times as strong as the root = 2.5 p. c. of colchicine. Parke, Davis & Co. (Drug. Bull., 89, 302).

Extractum Colchici Radicis Fluidum.

A good standard would be 1 p. c. of colchicine. Ten C.c., strongly acidified, should require 3.2 C.c. of Mayer's solution for complete precipitation. Parke, Davis & Co. (Drug. Bull., 87, 194). See also (Ibid., 89, 302).

Extractum Colchici Seminis Fluidum.

It is proposed to make the fluid extract of the seed of the same standard as the root, = 0.5 p. c. of colchicine. Parke, Davis & Co. (Drug. Bull., 89, 302).

Extractum Conii Alcoholicum.

A good standard would be 3 p. c. of coniine. Parke, Davis & Co. (Drug. Bull., 87, 195; 89, 303).

Assay. Similar to that under extract of aconite, but rendering the solution alkaline by potassa, and extracting the alkaloid with ether instead of chloroform. 1 C.c. of the acid indicates 0.00127 Gm. of coniine. Itallie (A. 89, 134. O. 89, 372. L. 89, 185).

Extractum Conii Fluidum.

A good standard would be 0.6 p. c. of coniine. Render the fluid extract alkaline, extract the alkaloid with benzin, shake with very

dilute hydrochloric acid (1 p. c.), and titrate with solution of phospho-tungstate of sodium. Parke, Davis & Co. (Drug. Bull., 87, 194; 89, 303).

Extractum Digitalis Fluidum.

Five C.c., evaporated to complete dryness at 105° C., should leave a residue weighing 1 Gm. Parke, Davis & Co. (Drug. Bull., 87, 194).

Extractum Ergotæ Fluidum.

Ten C.c., when mixed with 90 C.c. of alcohol, give a precipitate, which after drying at 100° C., should weigh 0.4 Gm. Parke, Davis & Co. (Drug. Bull., 87, 194).

Extractum Erythroxyli Fluidum.

A good standard would be 0.5 p. c. of alkaloid. Ten C.c., after removal of fatty, waxy and resinous constituents, should yield to ether, after addition of an alkali, 0.05 Gm. of alkaloid. Parke, Davis & Co. (Drug. Bull., 87, 194; 89, 303).

Extractum Euonymi.

Should be made with alcohol of not under 65 per cent. Naylor and Chaplin (N. 89, Dec. 473. B. 90, 22).

Extractum Gelsemii Fluidum.

Ten C.c. should require for complete precipitation 1.3 C.c. of Mayer's solution. Parke, Davis & Co. (Drug. Bull., 87, 194).

Extractum Glycyrrhizæ.

The U. S. Ph. does not state how to arrive at the weight of the soluble (or insoluble) portions. Hirsch (Un. Ph., II., 693).

Assay. Kremel considers the p. c. of glycyrrhizin a more reliable test of quality than the p. c. of soluble substance, because the commercial extract often contains other soluble extractive matter. He found, too, that the ash of genuine licorice has a strongly alkaline reaction, while that of the adulterated is generally neutral. He follows chiefly Diehl's method of assay (see G. 83, 31. O. 83, 55), the essential feature of which is to dissolve the extract in cold distilled water, to filter *after* the addition of alcohol, and to precipitate with diluted sulphuric acid. The precipitate is dissolved in ammonia and evaporated to dryness. He obtained from 5.88 to 11.00 p. c. of glycyrrhizin, and from 2.90 to 8.64 p. c. of ash (A. 89, 248. O. 89, 379. G. 89, 114. L. 89, 511).

Extractum Hyoscyami Alcoholicum.

Assay. Similar to that mentioned under extract of aconite. 1 C.c. of the acid indicates 0.00289 Gm. of hyoscyamine. Itallie (A. 89, 134. O. 89, 372. L. 89, 185).

Extractum Hyoscyami Fluidum.

A good standard would be 0.18 p. c. of alkaloid. Ten C.c. should require for complete precipitation 1.3 C.c. of Mayer's solution. Parke, Davis & Co. (Drug. Bull., 87, 194).

Extractum Ipecacuanhæ Fluidum.

A good standard would be 1.5 p. c. of emetine. Ten C.c. should require for complete precipitation 8 C.c. of Mayer's solution. Parke, Davis & Co. (Drug. Bull., 87, 194).

Standard. Braithwaite and Umney propose a standard fluid extract to contain 1.25 p. c. of emetine: Exhaust 100 parts of the coarsely powdered (No. 20) root with rectified spirit (0.838), reserve the first 80 fluidounces, then mix the marc with 10 parts of recently slaked lime, and exhaust again. Distil off the alcohol, and dissolve the extract in the reserved portion, adding sufficient rectified spirit to make a fluid extract of the desired strength. They find this way of making it preferable to mixing the lime with the root at the start (E. 89, 272. N. 89, Sept., 252. S. 89, Sept., 365).

Assay. The fluid extract is mixed with twice its volume of water, and the alcohol driven off on a water-bath. Filter, wash, and precipitate with subacetate of lead. Filter, remove excess of lead by hydrosulphuric acid, filter, render faintly alkaline with ammonia and shake with chloroform. Take up the alkaloid with acidulated water, and titrate with Mayer's solution. This is a modification of Ransom's method (Ibid.)

Extractum Malti.

Dieterich formulates the requirements of a good extract as follows: It should contain 60 to 67 p. c. of maltose; 2 to 4 p. c. of dextrin; sufficient diastase to convert 5 times its weight of starch into sugar; and 2 to 5 p. c. of albuminous substances (C. 89, 178. Helfenberg Annalen).

Estimation of Diastase. Percy Smith recommends as very exact to estimate the reducing power of the extract on Fehling's solution, both before and after the digestion of an excess of starch for 4 hours at 60°C. (A. 89, 432. S. 89, June, 873).

Estimation of the diastatic power. The differences existing in the requirements of a good extract (Dunstan and Dimmock require it to completely digest $\frac{1}{17}$ of its weight of starch in several hours; Jungk its own weight of starch within 10 minutes at 100° F.; Dymond $\frac{1}{8}$ of its own weight of starch in half an hour at 140° F.) are, according to Cripps, to be largely explained by the kind of starch used, since the several starches give widely differing results. 0.5 Gm. of an extract digested completely 0.05 Gm. of arrowroot in 5 minutes, while the same quantity of wheat starch was not quite digested in 45 minutes. Cripps formulates the requirements as follows: Extract of malt should completely digest its own weight of potato starch in 10 to 15 minutes at 98° to 100° F. He also gives a modified process of assay (N. 89, Dec., 481).

Extractum Nucis Vomice.

A good standard would be 15 p. c. of total alkaloids, more than one-half of which is strychnine. Parke, Davis & Co. (Drug. Bull., 87, 195).

Assay. Triturate with water acidulated with a few drops of sulphuric acid; after one hour precipitate with solution of acetate of lead (1:10), filter, add diluted sulphuric acid (1:25), filter, render

alkaline with ammonia, and take up with chloroform. Evaporate or distil off the chloroform, dissolve the residue in decinormal acid, and retitrate with centinormal soda. Itallie (L. 89, 184. Z. 89, 56). He found from 11 to 19 p. c. of alkaloids, and recommends therefore to put a lowest limit, which he thinks is better than a fixed p. c. of, for instance, 15 p. c., as recommended by Dunstan and Short (See Part I., 70).

Dieterich protests against the use of chloroform for taking up the alkaloids, because chloroform, on shaking it with the alkaline mixture, easily emulsifies and can only with difficulty be separated. Besides, it does not *completely* extract the alkaloids. He recommends an addition of alcohol to the aqueous solution, as advised by Beckurts (K. 87, 257), which prevents the emulsionizing (Z. 89, 98).

Dissolve 1 Gm. in 10 C.c. of water and 2 C.c. of dil. sulphuric acid (1.112) on a water-bath. Filter after cooling, render alkaline with soda solution, and shake, first with 20, then with 10 C.c. of chloroform; distil off the latter, and dry the residue at 100° C. The weight should be 150 Mgm.—15 per cent. If stronger add sufficient sugar of milk. If weaker, do not use the extract at all. Pharm. Neerl.

Extractum Nucis Vomice Fluidum.

A good standard would be 1.5 p. c. of total alkaloids. Parke, Davis & Co. (Drug. Bull. 87, 195; 89, 304).

Assay. To 10 C.c. add a few drops of diluted sulphuric acid, evaporate off the alcohol, wash the residue with ether, take up with water, render alkaline with soda, and extract the alkaloid with a mixture of ether and chloroform. (Ibid.)

Dilute 5 C.c. to 50 C.c. with water, add ammonia, wash out the alkaloids with chloroform, take up with diluted sulphuric acid, render alkaline with ammonia, take up with chloroform, evaporate to dryness and weigh. Snow (B. 89, 202. D. 89, 438).

Extractum Opii.

Process. Macerate 100 Gm. for 48 hours with 800 Gm. of distilled water, express strongly, and macerate the residue with 400 Gm. of distilled water for 24 hours. Mix the liquids, filter, and evaporate to dryness. The extract should weigh at least 50 Gm. It should contain at least 17 per cent. of morphine. Pharm. Austr.

Assay. Dissolve 3 Gm. of the extract in 42 Gm. of distilled water (cold), shake 30 Gm., of the filtrate with alcohol (0.830) 10 Gm., ether (0.725) 10 Gm., and ammonia (10 p. c.) 2 Gm.; collect the crystals, which separate within 24 hours at a temperature of 10 to 15° C., and wash them twice with a mixture of diluted alcohol (0.895), water, and ether (0.725), of each 2 Gm., and dry at 100° C. They should weigh at least 0.34 Gm. Pharm. Austr.

Process. Macerate 100 Gm. of powdered opium with 500 water for 24 hours, express, repeat twice with 300 water, each time for 12 hours, express, mix, filter, and evaporate to dry extract. Pharm. Neerl.

Assay. Rub 1.5 Gm. of the extract with 1 Gm. of slaked lime and 28.5 Gm. of water, macerate for not less than 12 hours, with frequent shaking, and filter. Shake 20 Gm. of the filtrate with 10 C.c. of ether and 5 drops of benzol, then dissolve in it 0.25 Gm. of

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Ferri et Quininæ Citras.

A test for tartaric acid would seem to be necessary. Brown has found it in some of the commercial salt (N. 88, March, 777).

Ferri Hypophosphis.

Assay. With permanganate of potassium; mercuric chloride; and with bromine. Moerck (A. 89, 326; 388).

Moerck finds that much of the commercial salt is insufficiently washed, and therefore contains sodium salts. He gives a process from calcium hypophosphite and solution of ferric chloride.—The U. S. Ph. test for ferric phosphate (insolubility in acetic acid) is unreliable. A better test is to boil the salt with an excess of an alkaline hydrate, filter, warm with hydrochloric acid, add a few drops of calcium chloride, and render slightly alkaline with ammonia; a flocculent precipitate indicates phosphite (A. 89, 389; 393).

Ferri Sulphas.

Atomic weight 277.457. Curtman (Meyer Druggist, 89, 153).

Test for purity. Dissolve 1 part in 25 parts of water, heat to boiling, oxidize with nitric acid and a little hydrochloric acid, precipitate by ammonia and filter. The filtrate should not be altered by sulphide of ammonium, nor after supersaturating with acetic acid, by ferricyanide of potassium. Krauch (B. 89, 92).

Test. Fennel prefers to dissolve a definite quantity of ferrous sulphate in an excess of sulphuric acid, which should require a definite quantity of the volumetric solution of potassium bichromate for complete oxidation, to be verified by ferricyanide of potassium (B. 90, 2).

Ferri Sulphas Praecipitatus.

Pour into "4" volumes of alcohol, which is kept rotating.—A solution of 1 part in 20 parts of recently boiled and cooled distilled water, should scarcely have any effect on blue litmus paper. Germ. Ph. Com. (L. 88, 1105).

Ferrum.

Vulpinus recommends ferrocyanide of potassium as the best general reagent for iron (K. 89, 599).

Ferric salts are rapidly reduced by percolating through metallic zinc, previously moistened with dilute sulphuric acid. Jones (B. 89, 180. K. 89, 378. Jour. Soc. Ch. Industry).

Test of identity. A solution of 5 grains of ferrocyanide and 1 grain of ferricyanide of potassium in a fluidounce of 20 p. c. alcohol is added to the acid liquid. Ferrous salts are detected by a solution of 263 grains of ferricyanide of potassium in 1 fluidounce of 20 p. c. alcohol. Meier (Drug. Bull., 89, 12).

Ferrous solutions are protected by hypophosphorous acid. Devine (B. 89, 150).

Ferrum Reductum.

Commercial iron by hydrogen has often an alkaline reaction, due to carbonate of sodium from the imperfectly washed hydrated oxide of iron. Beckurts, therefore, requires it not to alter the color of moistened red litmus paper at once (K. 89, 565).

Ipecacuanha.

Braithwaite and Umney propose to standardize all preparations by starting from a fluid extract containing 1.25 p. c. of emetine (A. 89, 529. E. 89, 273. N. 89, Sept., 252. S. 89, 365).

Geiger finds that diluted alcohol (0.890) is a better menstruum than wine, which latter ought to be replaced by the tincture (E. 89, 244. Z. 89, 551).

It seems to be more likely that the tannin of the wine combines with some of the emetine, precipitating it as insoluble tannate, and that therefore the wine of ipecac must always be weaker in emetine than the tincture.

Ipecacuanha contains a volatile alkaloid, the presence of which probably accounts for the greater yield of emetine sometimes obtained. Arndt (A. 89, 78. O. 89, 459. Ap. Zeit., 88, 1036).

Assay. Exhaust the finely powdered root with hot chloroform, containing 1 p. c. of ammonia (0.920), evaporate, dissolve in a known quantity of decinormal hydrochloric acid, filter, and retitrate with centinormal soda, using cochineal as an indicator. 1 C.c. of the decinormal acid indicates 0.0254 Gm. of emetine (X. II., 388).

Cripps and Whitby have critically compared the different methods of assay proposed (Flückiger; Lyons; Dragendorff; Zinoffski; Jones; Ransom) with the following results: 1. Lyons' process with ammoniated ether exhausts the root most fully, and is rapid of execution; they prefer, however, to use *cold* percolation, thereby avoiding so many weighings. 2. The ammoniated chloroform process of Flückiger is decidedly objectionable. 3. They strongly recommend the use of acetic ether, alone or with glacial acetic acid with or without chloroform. Exhaust the root with the menstruum, wash the percolate with slightly acidulated water; the aqueous solution is washed with ether while still acid, then, after being rendered alkaline with ammonia, with ether, and finally with chloroform. Evaporate to dryness (spontaneously and over sulphuric acid), dissolve the residue in water acidulated with sulphuric acid, and lastly titrate with Mayer's solution (half strength). 1 C.c. of the latter indicates 0.00945 Gm. of emetine. For practical purposes this process may be modified, so as to save time, and still give approximately correct results. They further controvert Flückiger's statement that the woody portion is practically inert. The whole root assayed 2.40 p. c., the woody portion amounted to 15 p. c. of the total weight of the root, and gave 0.8 p. c.; that is, about 5 p. c. of the total emetine existed in the woody portion (B. 89, 95. E. 89, 102. O. 89, 460. N. 89, Mch, 721).

According to the experience of Johnson the yield of emetine, as usually given (from 1 to 2.5 p. c.), is too low. Of 75 assays, representing 40,000 pounds, 5 gave an average of 4.15 p. c.; 6 over 3.5 p. c.; 22 over 3 p. c.; and of the remainder less than 6 gave under 2 p. c. The process followed was Dragendorff's, modified by Lyons. See (Drug Bull., 89, 355).

Jalapa.

Assay. 5 Gm. of No. 50 powder are mixed with fine sand, and percolated with 250 C.c. of boiling water. The dregs are digested with 150 C.c. of hot alcohol, filtered, and evaporated to dryness.

He seldom found more than 8.5 p. c. of resin. Drescher (A. 89, 340. B. 89, 156).

Kino.

An exhaustive investigation of the sources and physical properties of the different kinds of Australian (Eucalyptus) kino by Maiden, see (A. 89, 626. N. 89, Oct., 221; 321).

He divides them into 1, the *ruby* group, the chief characteristics of which are the formation of clear ruby solutions with alcohol and with cold water; and of a strongly gelatinous precipitate with acetate of lead. 2. The *gummy* group, scarcely soluble in alcohol, leaving a granular residue of gum. 3. The *turbid* group, so-called because the alcoholic solutions require a very long standing to become clear. The gelatinous precipitate by acetate of lead is slight. The best kinos for pharmaceutical purposes are the ruby group, if less than 1 or 2 years old; older ones gelatinize. The turbid group never gelatinizes.

Yield of ash. Breidenbach (A. 89, 70. O. 89, 487).

Krameria.

The root of *K. triandra* is distinguished from that of *K. Ixina* (New Granada) by the behavior of the aqueous maceration to ferric chloride, which produces a greenish coloration with the former and a black with the latter. On shaking 1 part of the root bark with 1 part of powdered iron and 300 parts of water, the liquid will after 4 hours have required a reddish brown color with the first-mentioned root, while the liquid from the last-mentioned root is colored violet (X. II., 390).

Lappa.

The yellowish decoction of the root is not rendered blue by iodine (X. II., 385).

Linum.

Ground flaxseed contains from 36.12 to 37.98 p. c. of oil. Puckner (A. 89, 442. B. 89, 151).

Liquor Acidi Arseniosi.

Provided that the liquid is perfectly alkaline, it will require 49.9 C.c. of the volumetric solution of iodine. Fennel (B. 90, 2).

Liquor Ammonii Acetatis.

It contains 7.85 p. c. of ammonium acetate (9.1 p. c. according to the extemporaneous formula). It is recommended to increase its strength 4 or 5 times, when it will keep better. Fennel (B. 90, 2).

Liquor Arsenii et Hydrargyri Iodidi.

Fennel gives a formula from the first ingredients:

Iodine, arsenious acid, corrosive sublimate, iodide of potassium, and water, for details of which see (B. 90, 3).

Nux Vomica.

On the relation of alkaloids to extractive matter. See Simonson (B. 89, 128. D. 89, 294).

According to the researches of Beckurts, the relative proportion of strychnine and brucine is subject to such variations, that preparations of Nux Vomica ought to be standardized on the basis of strychnine alone (E. 89, 276. K. 89, 574).

Assay. A good standard would be 2.5 p. c. of total alkaloids. Extract the finely powdered drug with Prolli's fluid (containing chloroform, see under Colchici Radix); separate the clear fluid, evaporate off the chloroform, acidulate with diluted sulphuric acid, and remove wax and resin by shaking with ether. Take up with a mixture of 1 vol. of chloroform and 3 vols. of ether, having previously rendered the liquid alkaline with soda. Separate the ethereal solution and evaporate. Parke, Davis & Co. (Drug. Bull., 89, 304).

Mix 10 Gm. of the powder with 10 Gm. of slaked lime and 3 Gm. of water, and exhaust with chloroform; evaporate to dryness, take up with 10 C.c. of decinormal hydrochloric acid, heat for a few minutes, filter, and retitrate with centinormal soda. 1 C.c. of centinormal acid indicates 0.00364 of alkaloids, assuming that strychnine and brucine are present in equal quantities. Beckurts (X. II., 423).

Should contain 2.5 p. c. of alkaloids: Percolate 5 Gm. of the powder with diluted alcohol (0.889), distil off the spirit and proceed as under Extractum Nucis Vomicae. If the powdered seed contains less than 2.5 p. c. of alkaloid, it should not be used; if more, dilute it with sugar of milk. Pharm. Neerl.

Marck recommends to rub the fine powder with about 9 times its weight of ignited sand before extracting it. As menstruum, he recommends to use a mixture of 3 parts of chloroform and 1 part of alcohol, to shake out with a 20 p. c. sulphuric acid, alkalinize with ammonia, and to take up with chloroform, which is evaporated or distilled off. (Z. 89, 57. Ned. Tijdschr.)

[Oleata.]

Behringer objects to the officinal process, because the oleates are not as permanent as when made by double decomposition. Instead of using a solution of castile soap (always of uncertain composition), he prefers a hydro-alcoholic solution of an alkali and a purified "red oil;" the presence of a small quantity of stearic acid he considers rather advantageous. The solutions of the metallic salts must be either neutral or slightly acid (A. 89, 593).

Oleatum Hydrargyri.

8 ounces of powdered castile soap and $3\frac{1}{4}$ ounces of bichloride of mercury are mixed with sufficient water to a pasty mass, which is thrown immediately into boiling water, and boiled until a yellow oily liquid is formed, which is washed, and heated until all the water is driven off. Brown (A. 89, 168. O. 89, 643).

See Parsons' objection to the process, followed by Brown. (Part I, 104).

Vulpus objects to Brown's process because by exposure to a prolonged heat (boiling water) more or less reduction of mercury

will take place. He recommends Dieterich's process, which is an improvement on that of the U. S. Ph. 25 parts of yellow oxide of mercury are triturated with 25 parts of alcohol, 75 parts of oleic acid added, the mass stirred until it begins to thicken, and after 24 hours the stirring continued at 60°C. until all the alcohol has been dissipated. The resulting mass is almost white, keeping so for several months. (K. 89, 601).

Behringer makes it from recently prepared oleate of potassium and solution of mercuric nitrate, at a temperature of 66° C. (A. 89, 598).

Harrison gives a review of the processes hitherto proposed. (N. 89, Nov., 443).

Oleoresina Aspidii.

The proper color of a well made oleoresin is greenish to brownish-yellow, but not grass-green, as so many commercial specimens are. Weppen and Lüders (Z. 89, 6).

Greenawalt finds the sediment more active than the supernatant liquid, and recommends to separate it from the oily layer. (A. 89, 169 O. 89, 379.)

[Olea Volatilia.]

Concentrated oils, and how far the essential oils are improved by concentration. Dieterich (G. 89, 113. Helfenberg Annalen).

Test for alcohol. Rice suggests to make use of de Koninck's test for alcohol in chloroform by means of permanganate of potassium (B. 89, 96).

Color reactions with phloroglucin. Ihl, see (A. 89, 180. O. 89, 590. Z. 89, 166).

Test for aldehyd with Nessler's reagent, see under Aether.

Test for chloroform. Treat the suspected oil with zinc and diluted sulphuric acid, and test the aqueous layer for hydrochloric acid with nitrate of silver (Oest. Ap. Ver. 88, 338).

Iodine absorption number of essential oils. The iodine absorption test is carried out differently by Barenthin, Kremel and Davies.

Barenthin: 0.1 to 0.2 Gm. of the essential oil is dissolved in 10 C.c. of chloroform, 25 C.c. (or an excess) of Hübl's iodine solution (a mixture prepared from 25 Gm. of iodine in 500 C.c. of alcohol, and of 30 Gm. of mercuric chloride also in 500 C.c. of alcohol, and filtered after 12 hours) added, and the whole allowed to stand for 3 to 4 hours. Now add 10 to 15 C.c. of a 10 p. c. solution of iodide of potassium, dilute with 150 C.c. of water, add a few drops of starch solution, and titrate at once with hyposulphite of sodium, until the liquid remains colorless for one minute.

Kremel: Dissolve the oils directly in the iodine solution, without the intervention of chloroform, and allow to stand 3 hours. Proceed as above, but continue the addition of hyposulphite of sodium solution until the liquid remains colorless for half an hour.

Davies: Dissolve the oil in chloroform, add an excess of iodine solution, and allow to stand for a whole night, when he proceeds in the same manner as Barenthin (see above). (A. 89, 301. O. 89, 591. E. 89, 126. N. 89, April, 821; 827.)

Snow: Follows Barenthin, but allows the mixture to stand for 40 to 48 hours before adding the solution of potassium iodide, etc. His

results show that the iodine absorption figures vary considerably according to the time during which the mixture is allowed to stand. For instance, oil of turpentine absorbs (for every 100 parts) in 3 hours 291; in 6 hours 333; in 15 hours 345; in 24 hours 353; and in 48 hours 397 parts of iodine (New Idea 89, March. N. 89, July, 4).

[*Olea Pinguia.*]

Hager removes rancidity by shaking 1 volume of the oil, warmed to 35° C. with 1¼ volumes of alcohol (85 to 87 p. c.) for 12 hours. This treatment is repeated twice with half a volume of alcohol (A. 89, 247. O. 89, 641. Z. 89, 192).

Test for mineral oil. Heat a piece of potassa (about the size of a pea) in a test tube with 5 C.c. of alcohol until dissolved, add 3 to 4 drops of the oil, boil for a minute and add 3 to 4 drops of water, when a turbidity will be observed. This test will show 1 p. c. admixture. Holde (A. 89, 413. Ap. Zeit. 89, 751).—Salzer makes use of liquefied carbolic acid (87 p. c. strength), which mixes clear with a certain definite quantity of oil, but already a few drops of mineral oil will render the mixture turbid (G. 89, 170. O. 89, 637. Z. 88, 724; 89, 360).

Nitrate of silver test. Brulle heats a mixture of 10 C.c. of the oil with 5 C.c. of fuming nitric acid, stirring briskly until froth appears, when different colors will be observed. On adding now 5 C.c. of an alcoholic solution of nitrate of silver (2½ p. c.), and heating to about 115° C., the silver salt will be decomposed, giving metallic reflexes of different colors. See (C. 89, 279. Comptes rendus).

Hübl's iodine number explained. Rice (B. 89, 80).

Oleum Amygdalæ Amaræ.

Test for nitrobenzol. Warm the oil with a little soda, add ferrous sulphate to destroy the odor of hydrocyanic acid, and finally a considerable excess of permanganate of potassium, which will oxidize the true oil to benzoic acid, while the odor of nitrobenzol remains unimpaired (A. 89, 77. L. 89, 127. Ch. Zeit. 88, 1727).

Oleum Amygdalæ Expressum.

The specific gravity should be put at 0.916 to 0.919; usually 0.918. —One part of the oil gives a clear solution, when heated in a water-bath with 4 parts of absolute alcohol; and with 34 parts of absolute alcohol in the cold. It requires 34 parts of boiling officinal alcohol for a clear solution.—A mixture of arachis and sesame oil, shaken with an equal volume of hydrochloric acid, becomes white, and the separated acid soon assumes a bright green color. Behringer, see (A. 89, 230. O. 89, 647).

10 C.c. of 87 p. c. carbolic acid should dissolve not less than 2.5 C.c., and not more than 3.5 C.c. of the oil without turbidity. Salzer (G. 89, 170. O. 89, 637. L. 89, 437).

Test for purity, based on the differing fusing points of the fatty acids. Almond oil becomes liquid at 14.2° C.; olive oil at 24 to 29° C.; sesame oil at 23.5 to 35° G.; cottonseed oil at 32 to 40° C.; arachis oil at 26.5 to 35° C. This test is reliable for 20 p. c. admixture. Ph. Germ. Com. (G. 89, 44. B. 89, 47. L. 88, 1111).

It is a non-drying oil. Does not congeal at 10°C . 4 C.c. of the oil, shaken with 4 C.c. of nitric acid (1.317), should not acquire a yellowish-brown or red color. On addition of a little mercury, the oil should, in a few hours, congeal to a nearly white mass. Pharm. Neerl.

A mixture of 15 parts of the oil, 2 of water, and 3 of fuming nitric acid, when shaken vigorously, should be white, not brown or red. Pharm. Austr.

Oleum Anisi.

The congealing points of the oils of anise and of star-anise have been determined by Umney, who distinguishes between the *true* congealing point—the temperature to which the thermometer immediately rises, on solidification taking place; and the *abnormal*, which is that usually given and determined while the oil is at rest. The abnormal congealing point of oil of star-anise is from 24 to 34°F .; that of oil of anise 50°F .; the true congealing point of oil of star-anise is from 49 to 56°F .; that of oil of anise 59°F . He thinks, however, that the best distinction between these oils is the behavior to a saturated solution of hydrochloric acid gas in absolute alcohol (Eykmán's test); oil of anise produces with this a manganese pink, and oil of star-anise a brown coloration. (A. 89, 255. O. 89, 600. N. 89, February, 647. S. 89, February, 229.)

Statistics by Schimmel & Co. See (A. 89, 311).

Congels at 9°C .; resumes fluidity at 15°C . Soluble in 2 vol. of alcohol (0.833). Pharm. Neerl.

Soluble in 3 parts of alcohol (0.830). A small piece of fused chloride of calcium, shaken with the oil, should neither liquefy nor get pasty. Pharm. Austr.

Oleum Aurantii Corticis.

Sp. gr. 0.850 to 0.870. Soluble in 7 vol. alcohol (0.833). Pharm. Neerl.

Oleum Bergamii.

The green color is, according to Schimmel & Co., always due to copper; the true color is honey-yellow with a tinge of green. A. 89, 312. O. 89, 593.)

Its common adulterants. Rizzuto (A. 89, 515. S. 89, Aug., 269).

Oleum Cajuputi.

Sp. gr. 0.920 to 0.930. Pharm. Neerl.

Oleum Cari.

Ph. Germ. Com. uses "carvol" with a boiling point of 224°C ., and a sp. gr. of 0.960.—A mixture of 10 drops each of oil and alcohol, is rendered faintly violet or reddish by 1 drop of solution of ferric chloride (1:100).—2 C.c. of alcohol mixed with 1 C.c. of water must dissolve at least 8 drops of the oil. (B. 89, 112. L. 89, 355).

Sp. gr. 0.910. Pharm. Austr.

Oleum Caryophylli.

On shaking 5 drops of the oil vigorously with 10 C.c. of lime water, a flocculent precipitate separates, partly adhering to the sides of the test tube.—2 drops dissolved in 4 C.c. of alcohol acquire a green color on the addition of 1 drop of ferric chloride; if the latter is diluted (1 : 20) the color is blue.—On shaking 1 Gm. of the oil with 20 C.c. of hot water, the latter should scarcely redden blue litmus paper. Ph. Germ. Com. (B. 89, 113. L. 89, 356).

Soluble in conc. acetic acid (1.06). Hot water, shaken with it, should not show an acid reaction, nor should the solution be rendered blue or green by a drop of solution of ferric chloride. Pharm. Austr.

Sp. gr. 1.041 to 1.060. Pharm. Neerl.

Oleum Cinnamomi.

Schimmel & Co. call attention to the fact that much of the oil of cassia is adulterated with resin and petroleum.—They formulate the requirements as follows: Sp. gr. at 15° C. 1.050 to 1.070. On distilling, about 90 p. c. of pure oil should pass over; the residue must not become solid and brittle, but remain semifluid, amounting to about 6 or 10 p. c. (A. 89, 370; 575. O. 89, 603. B. 89, 84).

Test for resin. The most reliable and easily applied test appears to be to ascertain the "acid" figure. Dissolve 2 Gm. of the oil in 30 C.c. of alcohol, add a few drops of phenolphthalein and titrate with potassa. Milligrams of potassa, for neutralizing 1 Gm. of oil, give the acid figure. Gilbert (A. 89, 609. Ch. Zeit., 89, 1406).

4 drops of the oil with 4 drops of fuming nitric acid should mix without effervescence, and produce a crystalline mass.—On shaking 1 drop with 10 C.c. of water, and adding solution of subacetate of lead, a white turbidity will appear, without the separation of yellow flakes (absence of oil of cloves.) Ph. Germ. Com. (B. 89, 113. L. 89, 357).

From cassia. 4 drops dissolved in 10 C.c. of alcohol, should not be rendered blue or green by a drop of solution of ferric chloride. Pharm. Austr.

From Ceylon cinnamon: Sp. gr. 1.020 to 1.040. Pharm. Neerl.

Oleum Fœniculi.

Sp. gr. 0.950 to 0.990. Soluble in half a vol. of alcohol (0.833). Pharm. Neerl.

Soluble in 3 parts of alcohol (0.830). Pharm. Austr.

Oleum Gaultheriæ.

Chemistry of the natural and artificial oil and of oil of birch by Trimble and Schroeter, who find that the oils of wintergreen and of birch are physically and chemically identical, and that the artificial oil loses its odor on the addition of an excess of potassa (A. 89, 398). Controversy regarding certain details of investigation by Powers and by the above-named authors, see (G. 89, 283. 90, 38. A. 90, 9).

Oleum Gossypii.

See olive oil for tests of identity.

Production in the United States. See (C. 89, 321).

Oleum Lavandulæ Florum.

Sp. gr. 0.885 to 0.895. Pharm. Neerl.

Oleum Limonis.

Soluble in 7 p. of alcohol (0.830). Pharm. Austr.

Soluble in less than 6 vol. of alcohol (0.833). Sp. gr. 0.840 to 0.855. Pharm. Neerl.

Oleum Lini.

10 C.c. of 83 p. c. carbolic acid should dissolve 3 C.c. of linseed oil. Salzer (G. 89, 170. O. 89, 637. L. 89, 441).

Oleum Menthæ Piperitæ.

An intense blue color appears on heating the alcoholic solution of the oil, to which a little sugar has been added, with hydrochloric acid (1.12) or dilute sulphuric acid. Ihl (A. 89, 180. O. 89, 590. Ch. Zeit., 89, 264).

Snow has investigated the tests for adulterations. Respecting the fuchsine test for alcohol he points out that less than 5 p. c. of alcohol would scarcely be added, and this would give a decided red color; light tints are to be disregarded, as being probably due to the presence of moisture.—Stevens' test for oil of camphor (Part II, 243) he recommends as very reliable, noticing, however, that some commercial oils assume a reddish-brown color on the addition of nitric acid. This color will disappear within 3 hours, while the reddish color of oil of camphor remains for 30 to 40 hours. It shows as little as 5 p. c.—As to the several tests for oil of turpentine, he warns against relying too implicitly on the rotation test, because French oil of turpentine rotates the ray of polarized light in the same direction as oil of peppermint.—The solubility in an equal volume of 80 p. c. alcohol (0.848) is not a very reliable test, because the oils of peppermint and turpentine are mutual solvents, so that an admixture up to 30 p. c. will be overlooked.—As to the iodine absorption test, the safest plan is to resort to double fractional distillation, and to compare the iodine number of the fractional part with that of the oil: the fractional part will show a much higher figure than the original oil.—The chloral-sulphuric acid test for oil of pennyroyal is unreliable, chiefly because the pure oil of peppermint gives too deep a color. (New Idea, 89, March. N. 89, June, 1056).

On dissolving 5 drops of the oil in 20 drops of glacial acetic acid, the impure oil soon acquires a dark-blue color with a cupreous lustre. Pharm. Neerl.

The oil mixes clear even with diluted alcohol (0.895). Pharm. Austr.

Oleum Morrhuæ.

Unger states that a good cod-liver oil should contain at most 4.5 p. c. of free fatty acid, and that when poured in a layer on top of nitric acid (1.40), after 5 hours, it will show a white ring on the line of contact (K. 89, 264).

10 C.c. of 83.3 p. c. carbolic acid should dissolve not less than 1 C.c. nor more than 2 C.c. of the oil. Salzer (G. 89, 170. O. 89, 637. L. 89, 443).

Morrhuc acid. Gautier and Mourgues (A. 89, 137. B. 89, 9; 32. C. 89, 318. Jour d. Ph. et de Ch.).

When cooled to 0° C., it should deposit no stearin, or but little of it. Pharm. Austr.

Oleum Myrciæ.

The sp. gr. of the pure oil is 0.970. It mixes clear with ether, benzin, chloroform and bisulphide of carbon; but not with alcohol and glacial acetic acid. It is perfectly neutral, and boils at 90° C. Mittmann (G. 89, 196. L. 89, 530).

Schimmel & Co. give the sp. gr. at 0.9806 (B. 89, 6).

Oleum Olivæ.

Test for cotton-seed oil. Labiche improves Bradford's subacetate of lead test as follows: Mix 10 C.c. of the oil with 10 C.c. of ether, add a concentrated solution of *neutral* acetate of lead, shake, and finally add 5 C.c. of ammonia, when an orange red color will indicate the presence of cottonseed oil. As little as 5 p. c. will be detected (N. 89, Sept., 242. Répertoire, 89, 352).

Wilson points out that, though the reaction with the nitrate of silver test proves the presence of cotton-seed oil, the absence of reaction does not prove the absence of this oil, because old oil does not reduce the silver. He considers Hübl's iodine absorption test as the most delicate and trustworthy. (A. 89, 195. O. 89, 638. Ch. News, 89, March, 99).

A chloroformic solution of auric chloride (1 Gm. in 200 C.c.) gives, on being heated with cotton-seed oil, an intense raspberry color, while pure olive oil gives no color reaction. Heat 3 to 5 C.c. of the oil with 6 to ten drops of the gold solution for 20 minutes on a water bath at 100° C. An addition of 10 p. c. cotton-seed oil can easily be detected, and even 1 p. c. with a little practice. Hirschsohn (A. 89, 23. B. 89, 143. O. 89, 639). Ph. Zeit. Russl. 88, 721).

Moerck, however, finds that this coloration is not peculiar to cotton-seed oil, but is also obtained with the oils of arachis, benne, poppy and sesame. He recommends a modification of Becchi's nitrate of silver method (A. 88, 65). He finds that age and exposure to light influence the reaction of both the silver and gold tests; he further criticizes the tests by sulphuric acid; nitric acid; elaidin reaction; "temperature" turbidity; "free acid" number; "saponification" equivalent; "iodine" absorption; and fusing point of fatty acid. See (A. 89, 225. O. 89, 639).

10 C.c. of 87 p. c. carbolic acid should mix clear with 2.5 C.c. of olive oil. Salzer (G. 89, 170. O. 89, 637. L. 89, 439).

On shaking 4 C.c. of the oil with 4 C.c. of nitric acid (1.317), the mixture should not acquire a reddish or brownish color. On adding to it a little mercury, it should congeal, in a few hours, to a nearly white mass. Pharm. Austr.

On mixing 15 p. of the oil, 2 of water, and 3 of fuming nitric acid, the mixture should become white, but not brown or red. Pharm. Neerl.

California olive groves. Searby, see (C. 89, 264).

California olive oil shows several deviations from the European standard. The sp. gr. is always higher (0.91747 to 0.91797); the

iodine reaction is longer delayed; the p. c. of free acids is much higher, instead of a fraction of one p. c., there is found from 1.09 to 5 p. c. and at times much higher, one sample showing 8.33 p. c.; the melting points of the acids are from 19 to 23° C.; the iodine absorption figure is from 88.09 to 88.69. Moerk (A. 89, 229. O. 645).

In confirmation of the above see also an article of Nagelvoort (Drug. Bull., 89, 357).

Oleum Picis Liquidæ.

Rowley states that much of the commercial oil is a solution of tar in rosin oil naphtha (A. 89, 235).

Oleum Ricini.

Test for rosin oil. Saponify, shake the solution with ether, separate the latter, and evaporate. With nitric acid (1.31), the color of the adulterated oil is decidedly dark, and the acid layer brownish; pure oil is colored light brown, and the acid remains colorless. Gilbert (A. 89, 611. Ch. Zeit. 89, 1428).

Test for coconut oil. Heat a sample, when the peculiar odor of coconut oil will be noticed. A mixture of equal volumes of pure castor oil and petroleum ether will always be turbid, but an addition of only 5 p. c. of another oil renders the mixture clear. Conroy (A. 90, 43. N. 89, Dec. 385).

Oleum Rosæ.

Method of examination by Schimmel & Co., who detect spermaceti by its being saponified by an alcoholic solution of potassa, while the stearopten of the oil of rose is not affected by the alkali (A. 89, 373. B. 89, 108. L. 89, 415).

At 22 C., the oil is soluble in 30 p. of alcohol (0.830) and the solution should not redden blue litmus paper previously wetted with water. Pharm. Austr.

Oleum Rosmarini.

Spec. grav. 0.880 to 0.913. Soluble in half its volume of alcohol (0.833). Pharm. Neerl.

Oleum Sabinæ.

Spec. grav. 0.920 to 0.950. Pharm. Neerl.

Oleum Santali.

Sp. gr.; iodine absorption; behavior to bromine, sulphuric acid, nitric acid, etc., of commercial oils. See Nagelvoort (Drug. Bull. 89, 357).

Spec. grav. 0.960. Pharm. Austr.

Oleum Sinapis Volatile.

A mixture of 4 drops of the oil and 25 drops of sulphuric acid should acquire no deeper tint than light yellow. Pharm. Austr.

Clean Theobroma.

Pharm. Neerl. In s p of ether. Pharm.

and shaken with a mixture of 5 parts of water and 1 of ammonia, it
 (b) of 100 parts of the mixture Pharm. Austr.

[illegible]

Oleum Tiglii.

Test.—A sample of sodium tetrachloro-iodate should be soluble in 2 volumes of water at 25° C. On vigorously shaking 2 volumes of the solution with 1 volume of fuming nitric acid and 1 volume of water, a yellow color should appear within 2 days. A mixture of 1 Gm. of the sodium tetrachloro-iodate and 10 Gm. of alcohol to which has been added 1 volume of water, iodine and 1 to 2 Gm. of powdered ferric chloride, should become decolorized within one hour; addition of 1 volume of 10 per cent. of iodine the reddish color should persist unchanged within one hour. Ph. Germ. Com. B. 89, 113. 14, 50.

the oil is added to the mixture with at least 15 C.c. of oil. Salzer

Opii Pulvis.

Preparation of this salt is indicated from opium dried at not above 100° C. (212° F.) for 24 hours. It should yield not less than 10 per cent of salt. If the opium contains more than 10 per cent of moisture, it should be dried as above directed. It should be mixed with enough water to make it to this percentage. Pharm.

Opium.

Alcali — Ether has been found that both the meconate and the sulphate of narcotine are soluble in chloroform, while the corresponding salts of morphine are practically insoluble, and concludes that chloroform is a better solvent and antagonistic for freeing opium from narcotine. Free narcotine requires only 2 parts of chloroform for solution. Meconate requires 1 part of chloroform.—It is highly probable that morphine is a constituent of opium as a *neutral sulphate* (L. 85, 205) the narcotine being there to a less extent as meconate. Possibly there is also a little meconate of morphine.—To make an assay of opium a sample is put in water that of barium as sulphate, he regards as an impurity, is added, exhausting the powdered opium with chloroform 20 parts with 10 times its weight of water, and set aside for 24 hours. On precipitating the morphine from the aqueous extraction by ammoniac, brown flakes of an extractive nature are separated, and the continuation can be prevented by the addition of alcohol, which, however, retains some of the morphine, the amount of which depends on the quantity of alcohol added. The less alcohol the more the mor. the more alcohol the purer the product. *Vol. 1, page 105.*—Another method, see Part I, 121 he finds that the more the narcotine is added, the more complete will be the separation, although the crystals will not be as

handsome as with less shaking. It is not difficult to determine the time when the morphine may be collected without the calcium salts.

To separate in *one* operation the *whole* of the morphine in a perfectly pure state is an impossibility.—His process, as now elaborated, may be formulated as follows:

Introduce 8 Gm. of powdered opium (of a degree of fineness to be fixed upon later) into a folded filter of 12 Cm. diameter, tapping the filter (to make the powder settle evenly), and dry the whole at 100° C. After half an hour, pour upon the powder a mixture of 10 C.c. of ether and 10 C.c. of chloroform and tap the funnel, which should be kept covered, frequently [to cause the solution to flow down]; finally add 10 C.c. more of chloroform. Cause as much of the fluid to drop off as possible, then spread out the filter and dry it, with its contents, at a very gentle heat. Transfer the powder to a flask, shake it (whether it has caked together or not) with 80 Gm. of water repeatedly and forcibly, and filter after two hours. (There is no objection to add to the water, previously, 0.2 Gm. of ammonium oxalate, but two hours will scarcely be sufficient to precipitate all the calcium.) Transfer 42.5 Gm. of the filtrate into a flask of known weight, add 7.5 C.c. of alcohol (sp. gr. 0.830), 15 C.c. of ether, and 1 C.c. of water of ammonia (sp. gr. 0.960), and shake repeatedly and briskly. After six hours transfer the contents of the flask upon two plaited filters, folded together, and of 10 Gm. diameter, and transfer as much of the morphine as possible upon the filter, by washing, using about 1.0 C.c. of water. Dry the filter first at a gentle heat, lastly at 100° C., and then transfer the morphine back into the flask, which has meanwhile also been dried at 100° C. Finally determine the total weight of morphine, when it remains constant at 100° C.

Flückiger also thinks that it might be worth while to ascertain whether morphine exists in opium really as a sulphate, or, at all events, whether there is a definite ratio between the morphine and the sulphuric acid. If such should be found to exist, it might be possible to determine the morphine by an estimation of the sulphuric acid. (B. 89, 191. L. 89, 721 to 732; 769.)

Nagelvoort modifies Flückiger's method as follows:

Ten Gm. of crude opium are dried at 100° C. for three hours, transferred to a dry mortar and reduced to a not too fine powder. A filter of 2 inches in diameter is filled with it (by "tapping" the funnel), and a mixture of 10 C.c. of ether and 10 C.c. of chloroform slowly passed through it, followed, when drained, by 10 C.c. of chloroform. When the last menstruum has passed, spread the filter out, and dry the contents. Transfer to a bottle holding about 120 C.c., add 100 C.c. of water, cork and shake frequently during 2 hours. Filter off 50 C.c., and shake at short intervals vigorously with a mixture of 10 C.c. of alcohol (0.820), 20 C.c. of ether and 1 C.c. of ammonia for six hours. Collect the separated morphine on a counterpoised filter with as little ice-cold water as possible (or with a saturated aqueous solution of morphine). Press the filter dry between blotting paper, and dry at 100° C. to constant weight. Multiply the result with 20, which will give the p. c. of morphine. (Drug. Bull., 89, 396.)

Dieterich criticises Flückiger's method, stating that chloroform does not free opium from all of its narcotine; that the use of alcohol must be avoided in methods of assay where the morphine is precipi-

tated by ammonia; and that the shaking method is too uncertain in its results. An opium, which assayed by Dieterich's own method (Part I., 122) 13.90 to 14.20 p. c. of morphine, yielded according to Flückiger's method, but without shaking, 3.18 to 6.55 p. c.; shaking every hour, 9.12 to 10.95 p. c.; shaking every half hour, 10.05 to 11.45 p. c.; finally, also by Flückiger's method, but without previous treatment with chloroform, 10.32 to 12.05 p. c. (K. 89, 582; 602.)

Pharm. Neerl.: Mix 3 Gm. of powdered opium with 1 Gm. of slaked lime and 10 Gm. of water. Add sufficient water to make a total weight of 32 Gm. Macerate for 12 hours at least, with frequent shaking, then filter. Shake 20 Gm. of the filtrate with 10 C.c. of ether and 5 C.c. of benzol, and dissolve in the solution 250 Mgm. of chloride of ammonium. Shake repeatedly. At the end of 24 hours, separate the ether; then repeat with 5 C.c. of ether. Collect the crystals; wash, first with water, then with 5 C.c. alcohol (40 p. c.), and dry at 100° C. They should weigh 0.2 Gm.

Pharm. Austr.: Macerate 8 Gm. of powdered opium with 80 Gm. distilled water for 12 hours. Mix 42½ Gm. of the filtrate with 12 Gm. of alcohol (0.830), 10 Gm. ether (0.725), and 2 Gm. of ammonia (10 p. c.), allow the solution to stand one day at 10° to 15° C., shaking it frequently. Transfer to a small filter, and wash the morphine crystals twice with a mixture of 2 Gm. each of diluted alcohol (0.895), ether and water; dry on the filter at 100° C. They should weigh not less than 0.4 Gm. The crystals, shaken with 100 parts of lime water, should, after several hours, form a yellowish solution, which color changes, on gradually adding chlorine water, to a brownish-red, and with ferric chloride to blue or green.

[Pepsinum.]

Tscheppé on pepsin, see (B. 89, 21. D. 89, 128).

Friedburg gives his process to free pepsin from chymosin (B. 89, 26. O. 89, 742).

Assay. Percy Smith prefers to use powdered albumen (B. 89, 26. O. 89, 744.)

Haigh tests as follows: Mix the pure pepsin with 19 parts of sugar of milk. Add to the saccharated pepsin, contained in a flask, 100 C.c. of a 2 p. mille hydrochloric acid, and 5 Gm. of albumen, which has been passed through a silk sieve of 120 meshes to the inch. Shake thoroughly, and place in a water bath at 38 to 40° C. for 3 to 4 hours, shaking every 15 minutes (Meyer Druggist, 88, 43).

Gibbons states that the proportion of available liquid in the stomach is probably not far from 1 ounce of water to each grain of pepsin in the gastric juice, and that, therefore, the same proportion should be used in tests (E. 89, 161).

Thompson has investigated the relative value of the different tests proposed:

Ph. B. prescribes 4.37 parts of 0.38 p. c. hydrochloric acid for 1 part of albumen at 130° F. for 30 minutes. U. S. Ph. directs 10 parts of 0.47 p. c. acid at 100° to 104° F. for 5 to 6 hours. In each case 50 grains of albumen to be digested by 1 grain of pepsin. The National Formulary directs 10 parts of 0.2 p. c. acid, 1 grain of pepsin to digest 500 parts of albumen at 125° F. in 60 minutes. Modified U. S. Ph. test: 10 parts of 0.30 p. c. hydrochloric acid for 1 of

albumen at 104° F. for 6 hours; 1 grain of pepsin to digest 50 grains of albumen. Thompson's proposed test: 10 parts of 0.2 p. c. hydrochloric acid (5.5 C.c. of acid of 1.16 to 994.5 C.c. of water) for 1 part of albumen, for 3 hours; 1 grain of pepsin to digest 500 grains of albumen. See for particulars and comparative results (B. 89, 168. O. 89, 112).

From Stutzer's researches it would appear that a previous treatment of albumen with ether or other fat-solvents will facilitate digestion and subsequent filtration (B. 90, March).

Preparation. Macerate rennet for 50 to 60 hours in water acidulated with hydrochloric acid, at a temperature of not over 50° F., precipitate with salt, dissolve in acidulated water, precipitate with alcohol, wash with diluted alcohol, and dry. This pepsin is claimed to dissolve 3000 parts of albumen. Scale pepsin is obtained by macerating rennet with dilute acetic acid for 5 to 6 days, evaporating it to syrup not over 105° F., and scaling. Schorp (C. 89, 269).

Petrolatum.

"Sulphuric acid of 1.540" is evidently a misprint for 1.840.

Pilocarpus.

A good standard would be 0.75 p. c. of pilocarpine, the maximum and minimum of a number of assays being 1.1 p. c. and 0.3 p. c. respectively. Parke, Davis & Co. (Drug. Bull., 89, 335.)

Assay. Exhaust the finely powdered leaves with water, add a little carbonate of magnesium, evaporate and filter. Shake with chloroform, which is separated and evaporated to dryness. (Ibid.).

Pilulæ.

Excipient for oxidizable pills. Macon replaces the paraffin of Martindale's excipient by spermaceti (A. 89, 467).

Pix Burgundicum.

Its solubility in alcohol should be given.

Pix Liquida.

A test for coal tar would seem necessary, because, according to Lowe, most of the commercial tar is a mixture of both. (A. 89, 159.)

Plumbi Acetas.

"Soluble in 1.8 parts of water."—Curtman points out the desirability of freeing the water from carbonic acid gas by previous boiling.—With litmus paper it should give an alkaline reaction. (Meyer Druggist, 89, 155.)

Plumbi Oxidum.

Test for its purity as a reagent. Krauch, see (B. 89, 115).

Tests for nitrous acid (as basic nitrates) and sulphate of calcium might be advisable, Sulzer having found both contaminations. (O. 89, 561. L. 89, 125. K. 88, 645.)

Test for sulphate of barium. Reichardt, see (L. 89, 753).

Podophyllum.

The Belgian Pharmacopœia requires it to contain from 3 to 5 p. c. of podophyllin.

It should contain about 4 to 5 p. c. of podophyllin, of which about 45 p. c. should be the podophyllotoxin of Podwyssotsky. Parke, Davis & Co. (Drug. Bull., 87, 194).

Potassii Bitartras.

Soluble in 192 parts of cold and in 20 parts of boiling water. Ph. Germ. According to Codex, in 250, and according to Ph. Norveg. in 170 parts of cold water.

“Absence of more than 6 p. c. of tartrate of calcium.”—Hirsch questions whether this is not a mistake for $\frac{1}{6}$ p. c. (Un. Ph. II, 799).—Ph. Germ. gives the test as follows: 1 Gm. of tartaric acid is digested with 5 Gm. of acetic acid (spec. gr. 1.041) for half an hour, diluted with 25 C.c. of water, filtered, and 8 drops of solution of oxalate of ammonium (1:20) added to the filtrate, when no alteration should take place within one minute (absence of more than $\frac{1}{4}$ p. c. of calcium tartrate).

Potassii Bromidum.

A test for barium might be advisable; it has been found several times by Güllich (Ch. Zeit. 89, 223. Ph. Zeit. Russl. 89, 554).

Potassii Chloras.

A test for lead would seem necessary. Krauch (B. 89, 92).

Potassii Cyanidum.

Its solution should not be colored red on addition of ferric chloride. Curtman (Meyer Druggist, 89, 254).

Potassii Hypophosphis.

Assay. With permanganate of potassium; mercuric chloride; bromine. Moerck, see (A. 89, 236; 391).

Potassii Iodidum.

Tests for nitrate. Boil the salt with sulphate of copper, sulphite of sodium and water, until all the iodine is precipitated as cupreous iodide, and test the filtrate for nitric acid in the usual way (sulphate of iron and sulphuric acid, or diphenylamine). Schwartz (A. 88, 612. Z. 88; 612. K. 89, 118).

Pour dilute hydrochloric acid over a piece of zinc in a test-tube, and add the solution of the salt, previously mixed with starch solution; a blue color indicates nitrate (Ibid.).

Heated in a loop of platinum wire, it imparts to the flame a tint which is violet from the beginning.

A better test for bromide would be desirable.

Test for iodate. Use recently boiled, distilled water, and after cooling add *immediately* the starch solution and the acid, otherwise there will be sufficient iodine liberated to produce a blue color. Ph. Germ. Com. (G. 89, 45. B. 89, 95).

Pyroxylinum.

The cotton should not be added before the temperature of the mixture has fallen to 20° C.—Wash finally with distilled water, until litmus paper remains unaffected, and dry at a temperature not above 60° C.—A moderately strong solution of bicarbonate of sodium expedites the removal of the acids. Meier (Drug. Bull., 88, 96).

Quininæ Bisulphas.

Dissolve 0.1 Gm. in 50 C.c. of distilled water; 5 C.c. of this should require, at most, 4 C.c. of ammonia for complete solution of the precipitate first formed. Keep protected from light (Pharm. Austr.).

Quininæ Hydrobromas.

Contains 76.6 p. c. of quinine. It loses at 100° C. not more than 5 p. c. of water. Soluble in about 60 parts of water. If it be dissolved in ammonia, the solution acidulated with diluted hydrochloric acid and mixed with a few drops of chlorine water, chloroform shaken with it should acquire a yellow color. Pharm. Neerl.

Dissolve 2 Gm. in 10 C.c. of warm water, add a solution of 0.5 Gm. of sulphate of potassium in 3 C.c. of warm water; evaporate to dryness; shake with 20 C.c. of water repeatedly during half an hour; heat to 60°–65° C., then cool and keep for 2 hours at 15° C., frequently shaking, and filter. 5 C.c. of the filtrate should give a clear mixture with 5 C.c. of ammonia (0.960) at 15° C. (Ibid.)

Quininæ Hydrochloras.

On dissolving 0.2 Gm. in 10 C.c. of distilled water with a few drops of nitric acid, it should require 5 C.c. of volumetric solution of nitrate of silver, so that after boiling and filtering, the filtrate be not sensibly opalescent after addition of either nitrate of silver or hydrochloric acid. The salt, moistened with solution of ferric chloride, should not be colored blue. Pharm. Austr.

Assay. Same as under Quininæ Hydrobromas, 5 C.c. of the filtrate requiring 5 C.c. of ammonia. At 100° C. it should not lose more than 9 p. c. of water = 81.7 p. c. of quinine. Pharm. Neerl.

Quininæ Sulphas.

Some criticisms on the tetrasulphate, bichromate, bisulphate and crystallization processes by Lenz, see (A. 89, 146. O. 89, 698. Jl. Ch. Soc., 89, 86).

Bromine water is recommended for titrating the alkaloids, based on the fact that a solution of sulphate of quinine requires considerably more bromine water to produce a permanent yellow color than a solution of any of the other alkaloids. Fawcett (B. 89, 111. O. 89, 694. N. 89, May, 914).

Examination of sulphate of quinine from 23 different sources by Paul and Cownley, showing variations in water of crystallization from 8.61 to 15.83 p. c.; in sulphate of quinine from 73.33 to 91.39 p. c.; in sulphate of cinchonidine from nothing to 12.34 p. c. (D. 89, 93. G. 89, 95. O. 89, 697. N. 89, Feb'y, 665.)

Snow has investigated the merits of Schäfer's oxalate method, using it as a quantitative test. See (M. 89, 481. O. 89, 698).

De Vrij has improved his chromate test as follows: 2 Gm. of the sulphate (or any other salt) are dissolved in 80 C.c. of boiling water, and to the clear and still hot liquid is added a solution of 0.55 Gm. of chromate of potassium (yellow). After being cooled down to about 15° C. (or lower), the separated chromate of quinine is collected on a filter, the mother liquor removed as much as possible by tapping on the funnel, and the crystals washed with a little water, so that the mother liquor and wash-water together amount to 80 C.c. Now add to the filtrate a few drops of solution of soda, or as much as will be distinctly indicated by phenolphthalein paper, when a turbidity or a precipitate will appear if other cinchona alkaloids are present. This turbidity will appear already while the liquid is cold, when the contamination is considerable; otherwise heating to 60° C. will be necessary. 2 p. c. will easily be indicated.—Schmidt recommends the chromate test as a very easy one, but advises to notice the turbidity which appears *immediately* after the addition of soda, because pure chromate of quinine itself separates within 12 to 24 hours. If the solution remains clear, even after having been heated to 80° C., less than 1 p. c. of other alkaloids is present. (K. 89, 291. Z. 89, 238.)

Ruddiman has examined into the merit of Kerner's test, especially elucidating the following points: 1. Comparison of results obtained by using for the initial digestion water at 100° C., followed by digestion at 15° C. with those obtained by digesting at once at 15° C. 2. The effect of variations in the temperature of the liquid during titration on the amount of ammonia necessary. 3. The accuracy of Kerner's figures for the amount of ammonia (0.920) required to dissolve 1 Mgm. of cinchonidine sulphate. 4. A proposed modification of the method of quantitative estimation of cinchonidine sulphate. 5. The amount of water which should be taken in applying Kerner's test to other salts of quinine. He sums up his results as follows:

1. The pharmacopœial test, when applied as directed, does not, as a rule, correctly indicate the amount of cinchonidine sulphate present. By using a higher temperature and sufficient water to dissolve all of the salt, a closer approximation to the real amount may be obtained.
2. The temperature at the time of titration has a considerable effect upon the amount of ammonia necessary to redissolve the precipitate. For every degree that the temperature is above 15° C., there should be added 0.148 C.c. to the number of C.c. of ammonia (0.920) used.
3. 0.36 C.c. of ammonia (0.920) is necessary to precipitate and redissolve 1 Mgm. of cinchonidine sulphate. In other words: Every 0.36 C.c. of ammonia used in titrating the 10 C.c. of filtrate from the sample being tested, over that used in titrating the 10 C.c. of filtrate from the standard quinine, indicates 1 Mgm. cinchonidine sulphate in 1 Gm. of the quinine sulphate to be tested, or 0.1 p. c.
4. In order to get a result nearest correct, it is necessary to dissolve all of the salt, which can be done by using 30 parts of water at 100° C.
5. The proportion of water to the sulphate in the U. S. Ph. test of other salts of quinine is not exactly the same as in Kerner's original test. There should be used for 1 part of the free alkaloid 11.7 parts of water; for the bisulphate 8.07 parts; for the hydrochloride 10.895 parts; for the hydrobromide 10.2 parts; and for the dried sulphate 10.144 parts. (E, 89, 18. Jour. Analyt. Chem).

Pharm. Neerl.: Heat 2 Gm. at 40° to 50° C. for half an hour, or

A good standard would be 0.3 p. c. of total alkaloids. Parke, Davis & Co. (Drug. Bull. 89, 304).

Tinctura Opii.

In order to prevent the deposit which sooner or later takes place, Ph. Russ. directs the addition of 0.5 Gm. of hydrochloric acid (sp. gr. 1.12) to every 100 Gm. of the recently prepared tincture.

Process. Opium, powdered, 20 Gm., alcohol (0.830) 90 Gm., water 150 Gm. Moisten with a sufficient quantity of the menstruum for 1 hour, transfer to a percolator, and percolate, after 48 hours maceration, to obtain 200 Gm. 100 Gm. of the tincture should contain the soluble matters of 10 Gm. of opium: about 1 Gm. of morphine. Pharm. Austr.

Opium, powdered, 10 Gm., diluted alcohol (0.889) 50 Gm., water 45 Gm. To make 100 Gm. of tincture. (Sp. gr. 0.975 to 0.977.) Pharm. Neerl.

Assay. Shake 40 Gm. of the tincture vigorously with 10 Gm. of ether and 2 Gm. of ammonia in a suitable closed container, and allow to stand for 1 day at 10° to 15° C. Collect the crystals, and wash them twice with a mixture of 2 Gm. of diluted alcohol (0.895), 2 Gm. of ether and 2 Gm. of water; dry at 100° C. The crystals should weigh at least 0.38 Gm. Pharm. Austr.

Evaporate 15 Gm. to 5 Gm., add 0.5 Gm. of slaked lime and 10 Gm. of water, so that the whole weight is 15.5 Gm.—Proceed as under Opium (see Pharm. Neerl.). The weight of the crystals should be 0.09 to 0.10 Gm.=0.9 to 1 per cent. Pharm. Neerl.

Tinctura Opii Camphorata.

Pharmaceutical assay. Drescher (A. 89, 338. B. 89, 155).

Tinctura Opii Deodorata.

Bateman modifies the process as follows: Percolate 10 parts of powdered opium with 28 parts of stronger ether, and, after drying the opium, digest it for 2 hours at 80° C. with 40 parts of water, which operation is repeated twice; mix the aqueous liquids, evaporate to 60 parts, filter and wash to obtain 80 parts; lastly add 20 parts of water (A. 89, 242. O. 89, 410).

Tinctura Vanillæ.

Quackenbush uses coarse sand, and considers prolonged maceration better than percolation (A. 89, 242. O. 89, 414).

Tragacantha.

Powdered tragacanth, shaken with chloroform, should, after one hour, not leave more than 1 p. c. of sediment (Ph. Zeit. Russl., 89, 70).

Maisch confirms his previous statement (P. 1548) about the precipitation of the mucilage by alcohol (A. 89, 72. O. 89, 657).—Ogle does the same (A. 89, 427. N. 89, July, 3).

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